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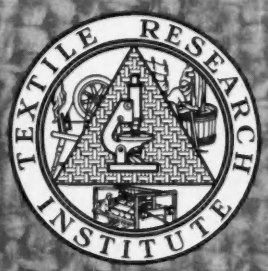

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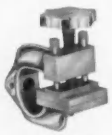

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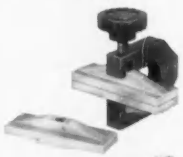

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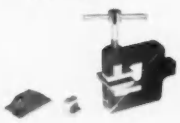

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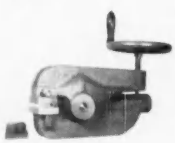

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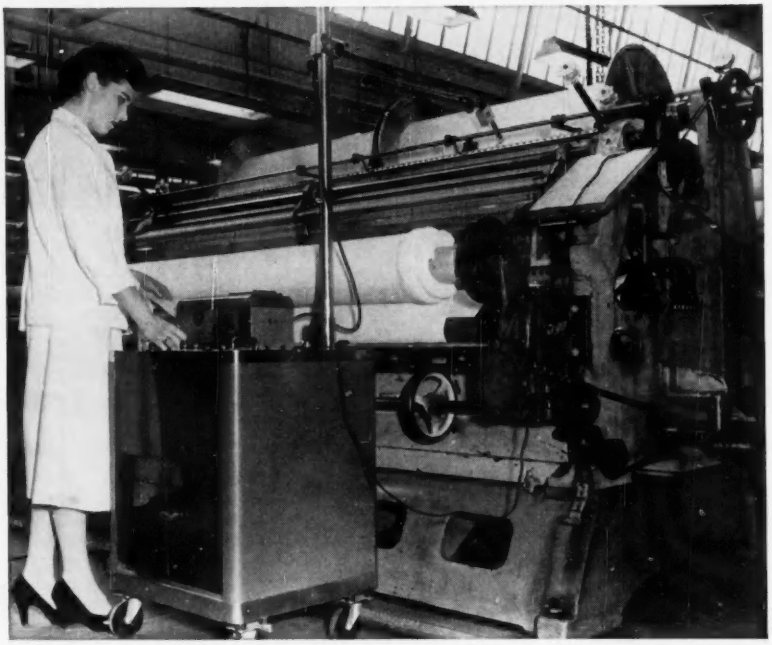

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TEXTILE RESEARCH JOURNAL

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Preparation of Microbiologically Resistant Wool by Chemical Modification[1]

Part III: Reaction with Monofunctional Compounds

Helmut Zahn and Albrecht Würz[2]

Chemical Institute of the University of Heidelberg, Heidelberg, Germany

Introduction

It was demonstrated in [11] that wool may be rendered permanently resistant to bacterial and fungous attack by means of treatment with chemically reactive compounds in such a manner as to combine functional groups of the wool proteins with definite molecular residues (chemical modification). The microbiological resistance was then determined by burial tests in active garden soil. Half-lives of up to 58 days were thus established for buried wool yarns.

Since a number of the chemically modified wool yarns proved to be so stable that they remained undecomposed during the burial time periods employed in the course of the investigations [11], it was found desirable to repeat the experiments and to test the samples with burial tests lasting several months.

Furthermore it was intended, in the present and in subsequent reports, to investigate which are the essential chemical modifications necessary for microbiological resistance. Thus, were the stabilizing effects produced dependent on the chemical constitution of the compounds employed or on the nature of the functional groups being modified within the wool proteins? Finally, which factors in the chemical treatment—concentration of the chemical compound, bath ratio, temperature, and duration of treatment, are of controlling influence for the effect?

In order to answer these questions, the methods of treatment found to be most promising in [11] were and will be repeated in present and subsequent research with appropriate variation of experimental conditions. In addition, new compounds were and will be employed and the reaction conditions for the methods found to give the best results were and will be studied in detail.

In this paper it is proposed to give an account of experiments with monofunctional acylating and alkylating reagents.

Description of Experiments

The Wool

For these experiments, the firm of Schachenmayr, Mann & Cie., Salach/Würtemberg, Germany, kindly placed at the authors' disposal samples of raw, white, noncarbonized "Sportgarn," spin No. 11½/4, N content 16.79%.

Reaction of Wool Yarns with Monofunctional Compounds

Group 1. *Acylating Reagents*

1. *Acetylation with boiling acetic acid anhydride.* After the method of Blackburn and Phillips [2].

2. *Acetylation with isopropenyl acetate* [4]. Air-dry wool (11.2 g) was heated to the boiling point in

[1] This paper appears in German in *Melliand Textilberichte*, February 1955. Part I of this series appeared in TEXTILE RESEARCH JOURNAL in September 1953; Part II in January 1954.

[2] Present address: Badische Anilin- & Sodafabrik, Ludwigshafen, Germany.

10 min with 200 cc isopropenyl acetate [3] and 0.2 cc concentrated sulfuric acid and then boiled under reflux for 30 min. The acetone formed in the course of the reaction was distilled off with the aid of a small fractionating column charged with Raschig rings, the distillate then being collected in a measuring cylinder. Acetone (16.5 cc) was found to distill over. The light yellowish-brown colored wool was sucked dry of dark-brown solution, washed with ethanol and water, and then dried in air.

3. *Acetylation according to the Casella process* [3]. Wool (12 g) was dried at 105° [4] for 1 hr and subsequently heated to 58° for 1½ hr with a mixture of 46.8 g acetic anhydride, 120 g glacial acetic acid, 4.5 g sulfuric acid, and 2.5 g diethylamine. The sample was sucked dry, washed with ethanol and water, placed overnight in a very dilute ammonia solution of *p*H 8.0, washed with distilled water, and finally dried in air.

4. *Benzoylation with benzoyl chloride.* Wool (11.2 g) was wetted with a solution of 10 g sodium bicarbonate in 280 cc water. Benzoyl chloride (7 g) was allowed to run in with mechanical stirring at room temperature. In the first hour there was a brisk evolution of CO_2 which gradually subsided. After 5 hr the sample was well sucked at the pump, washed with ethanol and water, placed overnight in a very dilute acetic acid solution of *p*H 4, rinsed with water, and finally dried in air.

It was possible to isolate 5.5 g benzoic acid from the acidified filtrate of the sample.

5. *Tosylation with toluene sulfonic acid chloride (tosyl chloride).* Wool (11.3 g) was treated with 6 g tosyl chloride and 3.6 g sodium bicarbonate in 240 cc acetone and 120 cc water and boiled for 3 hr under reflux. A *p*H value of 5.0 was obtained after cooling. The wool treated in this manner was sucked at the pump, washed with ethanol and water, and dried in air.

6. *Tosylation* as described in (5), using, however, 6 g bicarbonate instead of 3.6 g.

7. *Reaction with* p-*nitrobenzoyl chloride (in water).* Wool (11.3 g) was treated with 7 g *p*-nitrobenzoyl chloride and 10 g sodium bicarbonate in 300 cc water at 40° for 5 hr with mechancal stirring. After the termination of the moderate CO_2 evolution, the mixture was sucked at the pump, washed with ethanol and water, placed overnight in dilute acetic acid solution of *p*H 4, and then dried in air.

8. *Reaction with* p-*nitrobenzoyl chloride in acetone-water.* Wool (11.3 g) was treated for 5 hr at 40° with 7 g *p*-nitrobenzoyl chloride and 10 g sodium bicarbonate in 200 cc acetone and 100 cc water. In the first hour, a rapid evolution of CO_2 was observed. The wool thus treated was sucked at the pump and worked up as described in (7).

9. *Reaction with carbobenzoxychloride in water.* Wool (11.3 g) was treated for 5 hr at 0° with 10 cc freshly prepared carbobenzoxychloride and 10 g sodium bicarbonate in 300 cc water with particular attention to stirring. The reaction did not proceed uniformly. Purification was accomplished as described in (7).

10. *Reaction with carbobenzoxychloride in acetone-water.* Wool (11.4 g) was treated with good stirring for 5 hr at 0° with 10 cc freshly prepared carbobenzoxychloride and 10 g sodium bicarbonate in 200 cc acetone and 100 cc water. The N content of the carbobenzoxylated yarn amounted to 16.03%.

11. *Reaction with 4-fluoro-3-nitrobenzene sulfonic acid (FNBS).* Wool (11.5 g) was treated for 24 hr at 40° with continual stirring with a solution of 2.6 g potassium 4-fluoro-3-nitrobenzene sulfonate [12] and 2.5 g sodium bicarbonate in 300 cc water. Purification was carried out as described in (7).

12. *Reaction with 4-fluoro-3,3'-dinitro-diphenylsulfone (F-sulfone).* Wool (11.5 g) was stirred for 24 hr at 40° with a solution of 1.95 g of 4-fluoro-3,3'-dinitro-diphenylsulfone [12] and 2.5 g sodium bicarbonate in 200 cc acetone and 100 cc water. Purification was carried out as in (7). Millon's reaction was weak.

13. *Blank treatments for reactions 7, 8, 10, and 12.* Wool (11.3 g) was stirred with a solution of 2.5 g sodium bicarbonate in 200 cc acetone and 100 cc water for 24 hr at 40°. Purification was carried out as in (7).

Group 2. Alkylating Reagents

14. *Reaction with epichlorhydrin.* According to the method of Alexander, Carter, Earland, and Ford [1]. N content was 14.97%.

15. *Reaction with benzyl chloride.* Wool (10 g) was boiled for 5 hr under reflux with 4.95 g freshly distilled benzyl chloride and 5 g sodium bicarbonate in 200 cc acetone and 100 cc water. The yarn was

[3] Kindly supplied by Professor Dr. K. Hamann of Stuttgart, formerly of Uerdingen, Germany.

[4] All temperatures are in degrees centigrade.

sucked at the pump, washed with water, placed overnight in ethanol, sucked again, brought to *p*H 5 with acetic acid, and finally dried in air.

16. *Blank test for experiment 15.* Wool (10 g) was boiled under reflux with 2.5 g sodium bicarbonate in 200 cc acetone and 100 cc water for 5 hr and then purified as described under (15).

Results

Behavior of Untreated Wool in the Burial Test

In order to make a statistical examination of the reproducibility of the burial test using untreated yarn, 10 samples were each buried for a period of 7 days, after which the dry breaking strength and dry percentage elongation at breaking were determined. Table I shows that the mean standard error and the variation coefficient are comparatively low, especially when it is considered that a biological test method was employed.

The appearance of the wool samples was not uniform since bacterial attack takes place locally.

The Chemical Modification of Wool Yarn by Reaction with Monofunctional Compounds

Group 1. *Acylation.* Table II contains the results of the tests on yarns which have been acylated in a variety of ways. The table quotes the running test numbers of the chemically treated yarns the preparation of which is described in "Description of Experiments, Reaction of Wool Yarns," above; the half-life periods in days resulting from the burial tests; the solubility in alkali and in acid and the cystine content, which serve as chemical identity numbers; and finally the supercontraction in phenol. From the values in Table II it may be seen that wool

TABLE I. Reproducibility of the Burial Test with Untreated Wool Yarn *

	Individual Values (kg)					Mean	Mean Standard Error	Variation Coefficient (%)
Dry breaking strength, kg	1.90	2.21	2.42	2.07	2.34	2.18	0.32	15
	1.42	2.55	2.33	2.41	2.11			
Dry elongation at breaking, %	9.2	11.6	13.7	10.2	13.4	12.1	2.8	23
	7.1	16.0	12.7	15.5	11.7			

* Ten yarn tests, garden earth, burial period 7 days.

TABLE II. Properties of Wool Yarn Treated with Monofunctional Acylating Compounds

Sample No.	Treatment with	Half-Life Time (days)	Alkali Solubility (%)	Acid Solubility (%)	Cystine Content (%)	Supercontraction in 50% Phenol (%)
0	Untreated	11	12.0	12.0	11.55	49.7
1	Acetic anhydride	44	76.1	19.4	9.85	9.8
2	Isopropenyl acetate	14	17.9	13.9	13.3	46.2
3	Acetic anhydride (Casella)	14	56.0	16.3	10.0	10.4
4	Benzoyl chloride	11	13.4	9.8	10.85	35.4
5	Tosyl chloride	27	8.6	9.0	10.5	1.9
6	Tosyl chloride	42	4.6	12.7	5.2	7.9
7	*p*-Nitrobenzoyl chloride in water	11	12.7	9.2	11.35	37.1
8	*p*-Nitrobenzoyl chloride in acetone-water	11	9.2	10.3	11.35	7.2
9	Carbobenzoxy chloride in water	17	11.8	10.1	12.3	22.1
10	Carbobenzoxy chloride in acetone-water	15	9.5	8.6	11.4	8.2
11	FNBS	11	13.3	10.3	12.6	25.3
12	F-Sulfone	18	12.3	29.9	11.8	27.4
13	Blank (40°) to Samples 7, 8, 10, 12	16	13.6	13.6	11.8	50.0

TABLE III. Properties of Wool Yarn Modified by Treatment with Monofunctional Alkylating Reagents

Sample No.	Treatment with	Half-Life Time (days)	Alkali Solubility (%)	Acid Solubility (%)	Cystine Content (%)	Super-contraction in 50% Phenol (%)
0	Untreated	13	12.0	12.0	11.55	49.7
14	Epichlorhydrin	20	15.2	22.9	8.55	1.8
15	Benzyl chloride	17	8.3	21.3	5.2	12.5
16	Blank test, $NaHCO_3$ in acetone-water reflux	15	8.0	17.0	5.7	10.4

which has been boiled with acetic anhydride (Sample 1) under reflux possesses a half-life period of 44 days; i.e., it is capable of withstanding microbiological attack four times as long as untreated wool. In [11], a half-life period greater than 25 days was established. The reaction of wool with tosyl chloride (Sample 6) also proved effective. A half-life period of 42 days was achieved in this case. On considering the two best samples (1 and 6), it should be borne in mind that the acetylated wool (Sample 1) shows quite a high solubility in alkali, which points to an extensive change in chemical constitution. The tosylated wool (Sample 6), on the other hand, shows not only a high degree of biological resistance but also a low alkali solubility.

All other samples examined in the burial test for Table II were demonstrated to have been only moderately or not at all improved. This is probably due to the fact that the acid chlorides employed are saponified more rapidly than they are able to diffuse into the wool and react with the functional groups. The sulfonyl chlorides are hydrolyzed considerably more slowly, so that after diffusion has taken place the active compound is still present at a concentration sufficiently high to react with the wool. In our experiment, tosylation of the wool was achieved by boiling under reflux in a mixture of acetone and water. For technical purposes, this reaction would have to be carried out in an aqueous emulsion.

Group 2. Alkylation. Table III contains the results of the tests performed on alkylated wool samples.

In [11], a stabilization of wool by treatment with epichlorhydrin was recorded. Although an increase in resistance to soil burial was obtained, it was much less than that noted for acetylated wool (Table II, Sample 1). Alkylation with benzyl chloride gave only a slight effect. The slight stabilization achieved with the blank test (Sample 16 may have been because some of the cystine had, under very mild conditions, been converted to lanthionin. Stabilization was not only noted in the burial test but was also reflected in the increased resistance to alkalis and phenol.

Summary

The soil burial test for measurement of resistance of wool yarn to bacterial attack had a relatively high degree of reproducibility (variation coefficient of dry breaking strength is 15%).

The chemical modification of wool yarn with monofunctional acylating agents such as by treatment with boiling acetic anhydride solution or tosyl chloride in acetone-water produced wool resistant to fungous attack. Other chemical treatments proved to be either without effect or produced wool samples of lower resistance.

Alkylation treatments of wool with epichlorhydrin and benzyl chloride gave only slight improvement in soil burial resistance.

Part IV: Reaction with 1-Fluoro-2,4-Dinitrobenzene

Helmut Zahn and Albrecht Würz

Introduction

1-Fluoro-2,4-dinitrobenzene (FDNB) is an effective reagent for the preparation of wool resistant to fungous attack. As was reported in [11], dinitrophenylated yarns were found to be still intact after burial for 4 months. The present work was intended to investigate the influence of FDNB concentration and reaction temperature on the resistance.

Description of the Experiments

Group 1. *Reaction of Wool with FDNB in Ethanol-Water*

A description of the dinitrophenylation method has already appeared in [11]. Samples 1–7 were obtained in the same manner, using varying quantities of FDNB (Table I).

Group 2. Reactions of Wool with FDNB in Water, at Varying Concentrations

Quantities of materials used for this group, samples 8–13, are listed in Table I.

Group 3. *Reaction of Wool with* 1-*Chloro-2,4-Dinitrobenzene in Alcohol-Water at Varying Concentrations*

1-Chloro-2,4-dinitrobenzene was here employed in place of 1-fluoro-2,4-dintrobenzene. The quantities are given in Table II.

TABLE I. Quantities of Materials Used for the Preparation of Yarns Dinitrophenylated to Varying Degrees

Sample No.	Wool (g)	FDNB (g)	$NaHCO_3$ (g)
1	11.1	0.16	0.225
2	11.3	0.32	0.45
3	11.2	0.62	0.90
4	11.3	1.25	1.75
5	11.2	2.50	3.50
6	11.1	5.00	7.00
7*	11.3	—	7.00
8	11.3	0.16	0.25
9	11.4	0.32	0.45
10	11.3	0.65	0.90
11	11.2	1.25	1.75
12	11.4	2.50	3.50
13	11.2	5.00	7.00

* Blank test.

TABLE II

Sample No.	Wool (g)	ClDNB (g)	$NaHCO_3$ (g)
14	11.15	0.31	0.45
15	11.4	0.62	0.90
16	11.2	1.25	1.75
17	11.2	2.50	3.50
18	11.0	5.00	7.00

TABLE III

Sample No.	Temp. (°C)	Reaction Period (hr)	Wool (g)	FDNB (g)	$NaHCO_3$ (g)
19	60	4	4.0	0.5	1.0
20	60	4	4.0	1.0	1.6
21	60	2	4.0	2.0	3.2
22	60	4	4.0	2.1	3.2
23	80	2	4.0	2.1	3.2
24	80	2	4.0	4.0	4.0
25	80	4	4.0	2.1	3.2
26	100	1.5	4.0	2.1	3.2
27	40–100 100	0.5 1.0	4.0	2.1	3.2
28	40–100 100	0.5 1.0	4.0	2.1 ClDNB	3.2

Group 4. *Dinitrophenylation with FDNB at Varying Temperature* (60°–100°)

The preparation of the samples is listed in Table III. The quantity of water used in each case was 120 g.

Results

Group 1. *Reaction of Wool with* 1-*Fluoro-2,4-Dinitrobenzene*

Table IV contains the results obtained on yarns which were dinitrophenylated with increasing quantities of fluorodinitrobenzene in alcohol-water.

Table IV shows quite clearly that fluorodinitrobenzene stabilizes wool against microbiological attack only when a certain concentration of the compound is exceeded in the treatment. See Figure 1. Wool samples treated with dilute FDNB solutions are not stabilized. With increasing FDNB concentrations, the half-life period in the burial test increases progressively. The alkali solubility also increases correspondingly, while the supercontraction

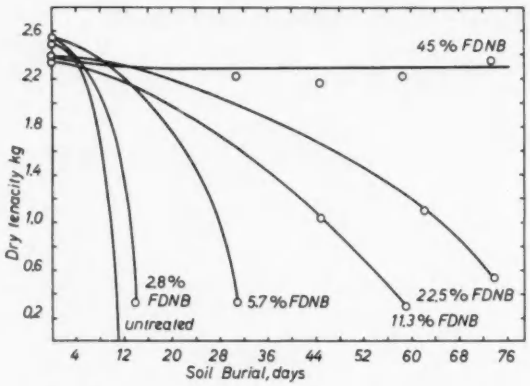

Fig. 1. Treatment of wool with 1-fluoro-2,4-dinitrobenzene (FDNB) in alcoholic-aqueous solution at 40° for 24 hr.

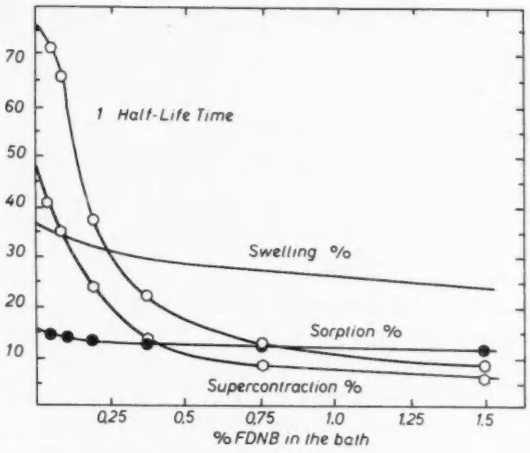

Fig. 2. Properties of wool yarn treated with 1-fluoro-2,4-dinitrobenzene in water at 40° for 24 hr.

and swelling decrease. The decrease in cystine content corresponds to a dilution of the wool protein by the dinitrophenyl residues which have been introduced. These experiments showed that the concentration of the reagents employed represents an important factor in the stabilization of the wool yarn against microbiological decomposition. The authors further recognize the possibility of being able to determine the degree of dinitrophenylation by means of a simple and rapid measurement of the superconcentration in phenol. The constant decrease in swelling appears to point to a relationship between accessibility of the fiber to bacterial attack and swelling. In this connection, the researches on acetylated cotton cited in [11] should be consulted.

Group 2. Fluorodinitrobenzene in Water

Alcohol as solvent is too expensive for the commercial application of dinitrophenylation of wool. Experiments were therefore carried out using water as solvent. As may be seen from Table V, a similar stabilization of wool with fluorodinitrobenzene may also be achieved by allowing the reaction to proceed in water-bicarbonate instead of in alcohol-bicarbonate. See Figure 2.

It may be seen from Table V that the changes in alkali solubility (increase) and of acid solubility, supercontraction, sorption, and swelling (decrease) run parallel to those obtained for dinitrophenylation in alcohol (Table IV). A certain agreement in the half-life periods for comparable amounts of reagent may also be detected in both Tables IV and V.

Group 3. Chlorodinitrobenzene in Alcohol-Water

The success of the experiments for protecting wool against bacterial attack by treatment with 1-fluoro-

TABLE IV. Properties of Wool Yarn Treated with 1-Fluoro-2,4-Dinitrobenzene in Alcohol-Water *

Sample No.	Quantity of FDNB with Respect to Wool (%)	Half-Life Period (days)	Alkali Solubility (%)	Cystine Content (%)	Supercontraction in 50% Phenol (%)	Swelling in Water (Centrifuge Values) (%)
0	Untreated	13	12.0	11.55	49.7	36.6
7	Blank test	11	8.5	10.90	46.1	35.6
1	1.4	11	9.8	10.85	39.1	36.6
2	2.8	11	10.8	—	32.1	34.6
3	5.7	26	11.9	10.55	21.6	32.2
4	11.3	43	15.7	—	—	30.0
5	22.5	60	19.5	11.10	14.8	28.3
6	45.0	>105	21.2	9.95	10.0	26.6

* 40°, bath ratio 30:1, 24 hr, 350 rpm.

2,4-dinitrobenzene led to the application of the less expensive 1-chloro-2,4-dinitrobenzene. Although in Samples 14–18 (no table for results is given) the same amounts of chlorodinitrobenzene were employed as in the series of samples 1–6, stabilization of the wool samples in the burial test was not obtained. A half-life period of 11 days was measured in all cases, the alkali solubility fluctuated between 9.2 and 11.7%, and the cystine content between 11.8 and 12.4%. The supercontraction also remained practically unchanged. These figures prove that dinitrophenylation of the wool does not succeed with chlorodinitrobenzene if this is allowed to act at 40°. Even though the wool had reacted with the chlorodinitrobenzene (the fibers had been stained yellow), most of the tyrosine phenolic groups had nevertheless failed to react. Experiments with chlorodinitrobenzene at a higher temperature showed that an exhaustive dinitrophenylation becomes possible by working at temperatures of at least 80°.

Group 4. Fluorodinitrobenzene at Higher Reaction Temperatures

A further investigation was carried out to ascertain whether it was possible to curtail the period of reaction by raising the temperature.

The results of tests on Samples 19–28 are listed in Table VI.

The results in Table VI demonstrate that it is indeed possible to shorten the period of dinitrophenylation very considerably by raising the temperatures employed from 40° to 60° to 80°. Sample 23 had been treated for only 2 hr at 80°, after which it still showed an undiminished dry breaking strength of 2.27 kg after burial for 140 days.

In dinitrophenylation the protective effect which can be imparted to the wool appeared to be due on the one hand to blocking, in particular of the tyrosine phenolic groups, and on the other to the introduction of residues with a higher molecular cohesion. The

TABLE V. Properties of Wool Yarn Treated with 1-Fluoro-2,4-Dinitrobenzene in Water *

Sample No.	Quantity of FDNB with Respect to Wool (%)	Half-Life Period (days)	Alkali Solubility (%)	Acid Solubility (%)	Cystine Content (%)	Supercontraction in 50% Phenol (%)	Water Sorption (% at 65% R.H.)	Swelling in Water (Centrifuge Values) (%)
0	Untreated	13	12.0	12.0	11.55	49.7	15.4	36.6
8	1.4	14	10.2	10.6	11.3	38.7	14.5	35.0
9	2.8	15	10.1	8.9	11.4	29.6	14.2	33.5
10	5.7	27	12.8	5.5	11.1	19.1	12.6	32.0
11	11.3	>46	14.3	4.7	10.5	13.2	13.1	30.0
12	22.5	>80	21.2	5.8	—	8.5	12.4	17.7
13	45.0	>80	—	4.6	10.1	5.6	11.2	23.9

* 40°, bath ratio 30:1, 24 hr, 350 rpm.

TABLE VI. Properties of Wool Yarn after Dinitrophenylation at 60–100°

Sample No.	Quantity of FDNB with Respect to Wool (%)	Temp. (°C)	Time of Reaction (hr)	Millon Reaction	Supercontraction in 50% Phenol (%)	Breaking Strength after Burial Period of (days)				
						32	60	88	105	140
19	12.5	60	4	Medium	12.1	Good	Good	Good	Good	Severely attacked (0.68 kg)
20	25	60	4	Weak	8.7	Good	Good	Good		
21	50	60	2	Weak	9.2	Good	Good	Good	Good	Good (1.72 kg)
22	50	60	4	—	9.6	Good	Good	Good	Good	Good (2.06 kg)
23	52.5	80	2	—	8.9	Good	Good	Good	Good	Good (2.27 kg)
24	100	80	2	—	13.9	Good	Good	Good		Good (1.75 kg)
25	52.5	80	4	Weak	18.7	Good	Good	Good	Good	
26	52.5	100	1	Weak	7.3	Good	Good	Good	Good	
27	52.5 CIDNB	100	1.5	Medium	13.4	Slight disintegration				
28	52.5	100	1	Weak	15.0	Good	Good	Good	Good	Severely weakened (0.80 kg)

obvious course of action was therefore to determine whether wool samples, which had been coupled with diazonium salts according to standard methods, had become more resistant in the burial test. It is known that the phenolic groups in tyrosine have a particular tendency to couple at positions 3 and 5. We allowed the diazonium salts of the following amines to react with wool: arsanilic acid, sulfanilic acid, *p*-phenylene diamine, benzidine, naphthylene diamine-1,5, *p*-chloro-*o*-methylaniline, 2-aminodiphenyl. Coupling took place at *p*H values between 9 and 9.5 and at a temperature between 0° and 10°, a reaction period of 20 to 30 min being employed. The coupled fibers were washed with very dilute acetic acid, sucked at the pump, and finally rewashed with distilled water. None of the samples so treated showed an increased life in the burial test. Thus no stabilization may be achieved by coupling. Solubility in acid of the coupled yarns fluctuated between 10.6 and 13.0 and, like the supercontraction in phenol (44.4 to 52.6%), remained practically unchanged. It has already been reported in the literature that wool cannot be protected against bacterial attack by coupling with diazonium salts.

Summary

Fluorodinitrobenzene proved to be a suitable reagent for preparing wool resistant to fungous attack provided that a sufficiently high concentration was employed. Using quantities of the fluoro compound amounting to 50% of the weight of wool, a resistivity of more than 100 days in the burial test was obtained at a bath utilization of 25%.

The same degree of success in protection against bacterial attack may also be attained with fluorodinitrobenzene in water instead of in alcohol as solvent.

Wool cannot be stabilized with chlorodinitrobenzene if the reagent is allowed to react at 40°.

A stabilization of wool with fluorodinitrobenzene as in second paragraph above, employing, however, much shorter periods of reaction, may be achieved by raising the reaction temperature to 60°–100°; at 100° the cheaper chlorodinitrobenzene also proved effective.

Part V: Reaction with Bifunctional Compounds

Helmut Zahn, Albrecht Würz, and Adolf Räuchle[5]

Introduction

As has already been demonstrated in [11], 4,6-(*bis*-chloromethyl)-1,3-dimethylbenzene was a reagent especially suited to the production of wool resistant to fungous attack since it had been possible to achieve both a long life in the burial test as well as a high degree of stability to alkali by virtue of the bifunctional mode of reaction with the wool proteins. It was therefore considered desirable to study in more detail this and other bifunctional compounds.

Description of the Experiments

1. *Reaction with* 1-*Fluoro*-5-*Chloro*-2,4-*Dinitrobenzene* (*FClDNB*) [10]

Wool (10 g) was treated for 14 hr at 40°, the motor operating at 200 rpm, with 5 g FClDNB and 7 g sodium bicarbonate in a mixture of 480 cc ethanol and 160 cc water. The yellow-stained wool was sucked at the pump, washed with alcohol and water, placed overnight in ethanol, brought to *p*H 4 with dilute acetic acid, and finally dried in air. N content was 16.06%.

2. *Reaction with* 1,5-*Difluoro*-2,4-*Dinitrobenzene* (*FFDNB*)

Analogous to Sample 1. FFDNB, kindly supplied by the Farbwerke Hoechst, Frankfurt-Hoechst, Germany, was employed in place of FClDNB. Millon's reaction very weak.

3. *Reaction with* 1,3-(bis-*chloromethyl*)-4,6-*Dimethylbenzene*

According to the method of Kirst [5, 6].

4. *Blank Test for* (bis-*Chloromethyl*) *Dimethylbenzene Wool*

According to the method of Kirst [5, 6]. Wool (10 g) was heated for 5 hr under reflux with a solution of 2.5 g sodium bicarbonate in a mixture of 250 cc acetone and 50 cc water [11].

[5] Present address: TAG, Krefeld, Germany.

5. *Reaction with* 1,3-(bis-*Chloromethyl*)-4,6-*Dimethylbenzene*

Wool (11.3 g) was boiled for 5 hr under reflux with 5.04 g *bis*-chloromethyl dimethylbenzene and 6 g sodium bicarbonate in 260 cc acetone and 130 cc water. Under these conditions the bicarbonate failed to go completely into solution. After the reaction had been completed, the wool was carefully washed with acetone and water, brought to *p*H 5 with dilute acetic acid, rewashed with water and acetone, placed overnight in ethanol, and finally rinsed and dried in air.

6. *Blank Test for Treatment with* bis-*Chloromethyl Dimethylbenzene*

Wool yarn (11.5 g) was boiled for 5 hr under reflux with 6.0 g sodium bicarbonate in 260 cc acetone and 130 cc water. The ol thus treated was sucked at the pump, thoroughly washed with distilled water, brought to *p*H 5 with dilute acetic acid, and finally rewashed and dried in air.

7. *Blank Test without Acetone*

Wool (11.2 g) was treated for 5 hr at 60° with 6 g sodium bicarbonate in 390 cc water. The sample was then worked up as described in (6).

8. *Reaction with* p-*Bromo-ω-Bromacetophenone According to the Method of Kirst* [5, 6]

Wool (10 g) was boiled for 5 hr under reflux with 5.82 g *p*-bromo-ω-bromacetophenone (kindly supplied by the Farbwerke Hoechst) and 2.5 g sodium bicarbonate in a mixture of 200 cc acetone and 100 cc water. The sample was then sucked at the pump, washed, placed overnight in ethanol, brought to *p*H 5 with dilute acetic acid, and then dried in air.

9. *Reaction with Benzal Chloride*

Wool (10 g) was boiled for 5 hr under reflux with 1.68 g benzal chloride [B.P. 88°–90° (12 mm)] and 2.5 g sodium bicarbonate in a mixture of 200 cc acetone and 100 cc water. The yarn was sucked dry, washed with water and ethanol, placed overnight in alcohol, brought to *p*H 5 with dilute acetic acid, and finally dried in air.

10. *Reaction with Benzotrichloride*

Wool (10 g) was boiled for 5 hr with 2.1 g freshly distilled benzotrichloride and 2.5 g sodium bicarbonate in a mixture of 200 cc acetone and 100 cc water. The yarn was sucked at the pump and then worked up as described in (9). N content was 16.62%.

11. *Reaction with* 4,4′-*Difluoro*-3,3′-*Dinitrodiphenyl Sulfone* (*FF-Sulfone*) [12]

Wool (11.5 g) was treated with a solution of 2.06 g FF-sulfone and 2.5 g sodium bicarbonate in a mixture of 200 cc acetone and 100 cc water at 40° for 24 hr with continuous mechanical stirring. The samples thus treated were then washed out with acetone and water, extracted for 20 min with acetone

TABLE I. Properties of Wool Yarns after Reaction with Polyfunctional Compounds

Sample No.	Reaction with	Half-Life (days)	Alkali Solubility (%)	Acid Solubility (%)	Cystine Content (%)	Super-contraction in Phenol (%)
0	—	13	12.0	12.0	11.55	49.7
1	FClDNB	130	10.0	4.0	9.74	—
2	FFDNB	150	8.5	3.6	11.3	3.4
3	*bis*-Chloromethyl dimethylbenzene (21%)	64	3.9	12.6	6.1	2.5
4	Blank test—acetone	11	8.3	16.0	5.5	18.0
5	*bis*-Chloromethyl dimethylbenzene (45%)	105	0	15.0	5.7	3.1
6	Blank test—acetone	—	—	—	5.4	8.4
7	Blank test—without acetone	11	9.6	13.6	12.4	44.3
8	*p*-Bromo-ω-bromacetophenone	>71	2.7	7.0	7.7	—
9	Benzal chloride	16	5.2	16.6	4.60	7.5
10	Benzotrichloride	26	12.3	7.3	11.50	3.6
11	FF-Sulfone	24	6.0	5.6	11.7	
12	FCl-Sulfone	20	9.8	5.1	11.4	16.9
13	FBr-Sulfone	21	13.6	4.2	11.1	12.5

in a Soxhlet, placed overnight in acetone, brought to *p*H 5 with dilute acetic acid, and dried in air.

12. *Reaction with 4-Fluoro-4'-Chloro-3,3'-Dinitrodiphenyl Sulfone (FCl-Sulfone)* [12]

Reaction analogous to (11), with 2.16 g of FCl-sulfone.

13. *Reaction with 4-Fluoro-4'-Bromo-3,3'-Dinitrodiphenyl Sulfone (FBr-Sulfone)* [12]

Reaction analogous to (11), with 2.43 g FBr-sulfone.

Results

Table I contains the data from tests carried out on the wool samples treated with the polyfunctional compounds described in "Description of the Experiments." From this table it may be seen that fluorochlorodinitrobenzene, *bis*-chloromethyl-dimethylbenzene, and *p*-bromo-ω-bromacetophenone stabilize wool best against the effect of soil burial. At first sight, the last compound might be expected to enter into a monofunctional reaction with the bromine in the aliphatic group. Since, however, both alkali and acid solubility have decreased markedly, the authors are of the opinion that this compound has reacted bifunctionally, although this assumption still awaits direct chemical proof. *bis*-Chloromethyl-dimethylbenzene, first introduced into wool chemistry by Kirst, proved to be particularly effective. If Kirst's method is modified, using instead twice the quantity of compound (Sample 5), the half-life period may be raised to 105 days. It is noteworthy that the alkali solubility of wool thus treated is completely eliminated. The authors also found the biological resistance to have been even somewhat enhanced subsequent to repeated washing. Since light and weather fastness are also good, the authors consider this compound to be the most promising of all those hitherto investigated by them. At present they are not in a position to advance a reaction mechanism theory for the *bis*-chloromethyl compound with wool. Model experiments (with E. R. Fritze) on N-benzoyl tyrosine point to reaction with the phenolic groups.

Benzyl chloride and benzotrichloride produce a wool of comparatively low stability.

The new halogen nitro derivatives, which were prepared by Zahn and Zuber [12], were also quite effective. Since the molecules are comparatively large, it was not possible to achieve complete blocking of the tyrosine phenolic groups or of the side chains in lysine under the conditions employed.

Summary

Reactions of wool with polyfunctional compounds demonstrated the effectiveness of fluoro-chlorodinitrobenzene, difluoro-dinitrobenzene, *bis*-chloromethyl-dimethylbenzene, and *p*-bromo-ω-bromacetophenone. It was also found here that the effect is dependent on the concentration of the reagents in the bath.

Part VI: Quinone, Ninhydrin, and Heavy Metal Salts

Helmut Zahn, Albrecht Würz, and Adolf Räuchle

Introduction

The protective action of quinone and of certain heavy metal salts has been variously reported in the literature [11]. It was therefore desirable to compare their effects on wool with those already obtained with compounds described in earlier communications. Ninhydrin, which had been recognized by Speakman [8] as a means of introducing cross-linkage into keratin fibers, was also included in this list.

Description of the Experiments

Group 1. *Treatment with Quinone and Ninhydrin*

Wool (12 g) was boiled under reflux for 1 hr with 360 cc of a 0.1–2% solution of benzoquinone in distilled water. The strands, which blacken during the course of the reaction, were washed in running water. The individual samples are listed in Table I.

6. *Treatment with quinone at 50° according to the method of Speakman* [7]. Wool (6 g) was

TABLE I

Sample No.	Concentration of Quinone Solution
1	0.1
2	0.5
3	1.0
4	1.5
5	2.0

warmed at 50° for 1 hr with 180 cc of a 1% aqueous solution of quinone (brought to *p*H 4.5 with acetic acid). Final *p*H value was 4.36.

7. *Treatment with quinone at* 50°. The same procedure as in (6), prolonged for 2 hr. Final *p*H value was 4.31.

8. *Treatment of wool with 0.075% quinone solution.* Wool (6 g) was heated at 50° for 1 hr with a solution of 90 mg quinone in 120 cc water (brought to *p*H 4.5 with dilute acetic acid). The bath was completely exhausted. Final *p*H value was 4.36.

9. *Treatment with ninhydrin, according to Speakman* [8]. Wool yarn (5 g) was boiled for 15 min with a solution of 2 g ninhydrin in 175 ml water, the solution being adjusted to *p*H 4.5. The sample was then sucked at the pump and washed thoroughly with distilled water. A cystine content of 10.32% was found.

Group 2. Treatment of Wool with Compounds Forming No Covalent Linkages with the Wool Proteins

10. *Impregnation with 2,4-dinitrophenol.* Wool (11.2 g) was placed for 2 hr at room temperature in a solution of 6 g of 2,4-dinitrophenol and 5 g sodium bicarbonate in 300 cc water. The yarn was then sucked thoroughly dry, placed for 10 min in 0.01 *N* hydrochloric acid, sucked once again, and washed.

11. *Treatment with mercuric acetate according to the method of Speakman* [9]. Wool yarn (11.6 g) was treated for 1 hr at room temperature with an 0.1 *M* mercuric acetate solution in 0.1 *N* acetic acid. The bath ratio was 30:1. The wool treated in this manner was sucked at the pump, washed with water, and dried in air. The N content of the sample amounted to 16.40%.

12. *Treatment with stannic chloride.* Wool yarn (11.5 g) was treated for 8 hr at room temperature with a solution of 32 g of $SnCl_4$ in 200 cc water. The sample was then sucked dry, washed, and dried in air.

13. *Chrome dyeing according to the Metachrom process.* Wool yarn (12.0 g) was dyed at a bath ratio of 30:1 with 4% (= 480 mg) Metachromrot 5G, 5% Glauber salt (= 600 mg), and 8% Metachrom mordant (= 1 g). Dyeing was commenced at 40°, the mixture was brought to boiling in the course of 30 min and then dyed by further boiling for 90 min. The yarn thus dyed was swirled thoroughly with lukewarm water and then adjusted to *p*H 5 with very dilute ammonia solution (*p*H 8).

14. *Blank chroming according to the Metachrom process.* Wool (12.0 g) was introduced at 40° into a solution which contained 5% Glauber salt and 8% Metachrom salt with respect to the wool employed. The mixture was brought to boiling within 30 min, and boiling was then continued for an additional 90 min. The bath ratio was 30:1. The chromed wool obtained in this manner was adjusted to *p*H 5 with dilute ammonia solution (*p*H 8).

15. *Chroming according to the afterchroming process.* Wool (12.0 g) was dyed with 4% Chromoxanbrillantrot BL (Farbenfabriken Bayer) and 10% Glauber salt. Dyeing was begun at 40°, the solution was brought to boiling in the course of 15 min, 2% sulfuric acid was added, and boiling was continued for an additional 30 min. The yarn was removed, the bath cooled down to 30°, 2% potassium dichromate added, the sample then being boiled for another 45 min. The yarn was finally removed, rinsed thoroughly with lukewarm water, and adjusted to *p*H 5 with dilute ammonia.

16. *Blank dyeing according to the afterchroming process.* Treatment similar to (15) without dyestuff and with the addition of 5% acetic acid to which 10% Glauber salt was added.

Results

Group 1. *Treatment with Quinone and Ninhydrin*

Table II contains results of tests carried out on yarn samples treated with quinone.

The large decrease in alkali and acid solubility and in supercontraction give proof of a reaction which has led to the formation of crosslinkages. It may be inferred from the decrease in total nitrogen

content from 16.5 to 13.5% that the wool proteins have taken up approximately 20% quinone. Accordingly, the bath had only been one-third exhausted. This gives a renewed confirmation of the fact that the most important factor in producing a resistant wool is a sufficiently high concentration of reagent. A boiling 0.5% quinone solution is sufficiently concentrated to stabilize wool and increase its life very markedly. At the time of writing, Samples 3–5 had still not undergone rotting. As a result of earlier preliminary experiments (Samples 6–8), it was shown that even a 1% quinone solution is incapable of giving sufficient protection at a reaction temperature of 50°, whereas boiling for 1 hr in a 1% quinone solution (final *p*H-value 4.35) leads to a product exhibiting an undiminished breaking strength of 2.89 kg after burial for 112 days.

A similar reaction with a compound containing carbonyl groups is that of ninhydrin with wool at *p*H 4.5, which was first investigated by Speakman. According to Speakman [8], ninhydrin is a most effective reagent for introducing crosslinkages into wool. On investigation of a wool sample according to Speakman's method, the authors were able to establish a considerable degree of stabilization in the burial test (cf. Sample 9, Table II).

Group 2. Testing Yarn Samples Treated with a Variety of Compounds

Table III contains a list of the results of tests carried out with yarns which had been impregnated or reacted with various compounds. Impregnation of the wool with dinitrophenol led to a marked stabilization which was, however, far less than that achieved by dinitrophenylation (cf. Part IV). Reaction of wool with mercuric acetate, according to Speakman's method, also proved effective. Treatment with stannic chloride was, on the other hand, not successful. Blank chroming, using the Metachrom process, led to a marked degree of stabilization. The same, but to a still greater extent, also applied to wool which had been afterchromed. In this case, the authors observed a whole life resistance of 75 days. Although much work has already been carried out on the influence of chroming on biological resistance, further investigation, especially with differing amounts of chroming agent, are still considered to be necessary.

Summary

Treatment with a quinone solution of minimum concentration 0.5% at 100° leads to a high degree of stabilization of wool against fungous and bacterial attack. Reaction with ninhydrin (Speakman) is also very effective.

Treatment of wool with compounds which undergo salt or complex formation with the wool proteins gives the following results: Impregnation with dinitrophenol leads to marked stabilization as also does reaction with mercuric acetate and various chromic processes.

Chemical modification commands practical interest if, in addition to stability against bacterial attack, further technically desirable effects, such as resistance to attack by acids, alkali, and moths can also be imparted. Dinitrophenylation renders protection against bacteria and, to a certain, degree, also against moths. Treatment with *bis*-chloromethyl benzene protects against attack by bacterial, moths and alkali.

Addendum

Possibilities for Commercial Applications

One might well be of the opinion that the practical importance of the chemical modification of wool as a means of protection against bacterial attack is not

TABLE II. Properties of Wool Yarn after Treatment with Quinone and Ninhydrin

Sample No.	Treatment with	Life Period in Burial Test (days)	Alkali Solubility (%)	Acid Solubility (%)	Super-contraction in Phenol (%)	N (%)
1	0.1% quinone soln.	16	8.5	10.1	6.5	—
2	0.5% quinone soln.	101	10.7	2.9	0.5	15.55
3	1.0% quinone soln.	>230	8.7	6.2	0.5	14.70
4	1.5% quinone soln.	>230	10.3	7.2	1.0	14.01
5	2.0% quinone soln.	>230	13.8	4.8	4.0	13.08
9	Ninhydrin	204	25.0	9.25	—	13.88

TABLE III. Properties of Wool Yarns after the Action of a Number of Organic and Inorganic Reagents

Sample No.	Treatment	Half-Life Period (days)	Alkali Solubility (%)	Acid Solubility (%)	N (%)	Cystine Content (%)	Super-contraction in Phenol (%)
0	Untreated	13	12.0	12.0	16.15	11.30	49.7
10	Dinitrophenol	28	13.5	16.0	—	11.75	8.2
11	Mercuric acetate	45	21.7	15.4	—	9.16	13.4
12	Stannic chloride	11	18.0	14.5	15.18	10.46	11.2
13	Metachromrot dyeing	37*	10.6	14.5	15.86	11.05	24.5
14	Blank chroming (Metachrom process)	33*	9.3	16.2	16.01	11.00	23.8
15	Afterchroming with Chromoxanbrillantrot BL	67*	14.3	17.5	15.83	10.94	8.3
16	Blank chroming (afterchroming process)	75*	19.6	12.6	15.58	10.50	6.1

* Whole life time.

particularly great since the chroming of wool already provides a comparatively cheap method of treatment.

Further objections may be raised against chemical treatment with relatively large quantities of reagents which not only alter the texture of the wool but also demand the use of special methods not normally employed in the treatment of wool. The authors had a number of very enlightening discussions concerning this point with colleagues from the textile industry and should, in passing, like to take this opportunity of expressing our sincere thanks to them.

The authors believe, however, that it should be possible to discover a practicable means of chemical modification which requires no specialized method of treatment. The successful dinitrophenylation, for example, where this could be carried out in water instead of in alcohol in 2 hr, was especially promising. Even though dinitrophenylation itself could hardly be employed technically, owing to the low degree of fastness to light and the color of the material so produced, the experiments nevertheless do demonstrate the possibility, in principle, of working under conditions comparable with those operating in technical practice.

As long as no experimental data are available on the handle, behavior on felting and milling, dyeing properties, etc., it is not possible to make any statement concerning the practical suitability of the individual method. The authors believe, however, that chemical modification will not gain any exalted practical importance until a method of producing an enhancement of several properties by *one* single process has been successfully worked out. Thus, in preliminary experiments by Dr. Laibach of Farbenfabriken Bayer Leverkusen, wool treated with acetanhydride, fluorodinitrobenzene, difluorodinitrobenzene, *bis*-chloromethyl-dimethylbenzene, and quinone proved to be stabilized against attack by moths. Furthermore, it may be gathered from the authors' work that numerous methods of treatment not only result in an increase of life in the burial test but also diminish alkali and acid solubility.

When it is considered that a decrease in supercontraction in phenol is a result of an increased thermal stability, it is possible that wool samples treated in the appropriate manner would be more resistant to high-temperature dyeing. A high degree of stability to alkali might be of use for dyeing with vat and naphthol dyes. Bleaching with peroxide is also rendered less detrimental by reducing alkali solubility before treatment, as, for example, is already in practice in the new Du Pont formalization process. The authors therefore intend to continue work in various directions.

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A Study of Cotton Fiber Perimeters as Calculated from Arealometer Values at Low and High Compression

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Abstract

For a group of cottons selected to show a wide range in fiber perimeter, differences in perimeter as calculated from arealometer specific surface and D, for ginned lint samples, were similar to differences in primary-wall perimeter; and the actual perimeter values differed little from those calculated from specific surface and array weight per inch. Moreover, calculated weight per inch differed little from values obtained by the array method. It was shown for a second group of cottons that, with but few exceptions, D, and consequently perimeter, etc., calculated from specific surface and D, decreased from raw stock to picker lap and then increased successively for card sliver, first and second drawing sliver, and roving, the greatest changes in D occurring in the conversion of card sliver to first drawing sliver and second drawing sliver to roving. These results indicate that the fiber property values calculated from A and D will be valid for ginned lint samples, but will vary with processed samples and particularly will not be valid for samples that have been subjected to considerable mechanical drafting.

Introduction

The *general* importance of fiber perimeter as one of the properties affecting yarn quality has long been recognized, since perimeter, along with the amount of secondary-wall development, determines "fineness" or the number of fibers that will occur in the cross section of a yarn and therefore determines, in part, yarn strength.

It is well known that long and fine cottons will make stronger yarns than short and coarse ones; definite information is lacking, however, regarding the specific importance of the effect of perimeter alone, dissociated from fiber length and wall thick-

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ness, on yarn quality and spinning behavior. This lack is due in part, at least, to the fact that there has been no rapid yet accurate means for measuring perimeter alone, and thus there is no large accumulation of data from which to draw conclusions as there is for fiber length, for example, or fiber strength. For the accumulation of such perimeter data the measuring of individual fibers is too tedious, while the measurements obtained by the use of any of the air-flow instruments [1, 2], though rapidly made, reflect both perimeter and the amount of secondary-wall development, and, as such, are not satisfactory [1, 2, 4]. A recent modification of the arealometer [8] offers promise, however, of fulfilling the need for a rapid method of determining perimeter [3]. According to this method 2 values are obtained, 1 with low compression (high porosity) and 1 with high compression (low porosity); the difference between these two is D, which is related to the amount of flattening of the fibers or the amount of immaturity. Immaturity ratio, I, is calculated from D, and I is used with specific surface, A, to calculate perimeter, weight per inch and wall thickness.

Arealometer data obtained over a period of three years in connection with the spinning program of the U. S. Department of Agriculture, Section of Cotton and Other Fiber Crops and Diseases [9, 10, 11], and other observations made in connection with related studies have indicated, however, that the D value may be influenced by interfering factors. For example, the D value for second drawing sliver was found to be almost invariably larger than that for the ginned lint of the same cotton [9, 10, 11] with the result that the calculated values of perimeter, etc., would not be the same for the two different materials. The actual occurrence of such alterations of fiber properties with processing does not seem probable. The need for a critical study of perimeter values as calculated from arealometer data is thus apparent. This paper presents the results of a study in which, for a series of cottons representing a wide range in specific surface, comparisons were made among perimeter values obtained in three different ways. Evidence is presented to show that for comparative purposes the reliability of perimeter values calculated from A and D may be affected by differences in the maturity of the cottons and type or amount of manipulation the lint received prior to testing.

TABLE I. Cottons for Which Perimeter Measurements Were Made, Listed According to Increasing Primary-Wall Perimeter

Code number	Cotton	Place of growth	Year of growth
1	S×P	Sacaton, Ariz.	1948
2	Karnak	Sacaton, Ariz.	1948
3	Sea Island (selection)	John's Island, S. C.	1947
4	Sealand 542	Florence, S. C.	1947
5	Triple Hybrid 805×6	Raleigh, N. C.	1946
6	Acala 1517 W	Florence, S. C.	1947
7	AHA-4-1-76-184	Shafter, Calif.	1949
8	Acala 1517 W	Raleigh, N. C.	1947
9	Sealand	Raleigh, N. C.	1947
10	AHA-46-124-52	Shafter, Calif.	1949
11	Sealand 542	Raleigh, N. C.	1946
12	Wilds	Florence, S. C.	1947
13	Sealand 542, Off-Type	Florence, S. C.	1947
14	Acala 1-1-1-2	Raleigh, N. C.	1946
15	Acala 1-1-1-3	Raleigh, N. C.	1946
16	Stoneville 2B	Shafter, Calif.	1949
17	Rowden 10-5-2-1	Raleigh, N. C.	1946
18	Stoneville 2B	Raleigh, N. C.	1947
19	Deltapine 15	Raleigh, N. C.	1947
20	Deltapine 15	Shafter, Calif.	1949
21	Station C	Raleigh, N. C.	1947
22	Oklahoma Triumph	Raleigh, N. C.	1947
23	Stoneville 2B, Doubled Haploid	Raleigh, N. C.	1946
24	Rowden 18-2-1-4	Raleigh, N. C.	1946
25	Rowden 40-5-3-1-2	Shafter, Calif.	1949
26	Rowden 40-5-3-1-2	Raleigh, N. C.	1946
27	Acala 36	Raleigh, N. C.	1946

Materials and Methods

Twenty-seven cottons were used as a basis for comparing perimeter values obtained by three different methods (Table I). These cottons represented four different years of growth, five locations, and a wide range in staple length and perimeter. A few varieties were grown at more than one location and in more than one year. On 9 of the cottons a study of perimeters had been made previously [7].

The three methods of determining perimeter were: (*1*) Measurement in the primary-wall stage—27 cottons; (*2*) calculation from arealometer A and D—26 cottons; and (*3*) calculations from specific surface A and array weight per inch—19 cottons. Each of these methods is described separately in detail.

To obtain the materials on which these measurements were made, approximately 50 blooms of the selected cottons were tagged with dated tags. When the young bolls were 10–18 days of age some of them were collected and the young cotton locks boiled to separate the fibers and then preserved in 70% alcohol [7]. This material was used in making measurements of the perimeter of the primary walls. The remaining bolls were allowed to mature and

provided the lint for arealometer and weight-per-inch determinations.

To facilitate the presentation of data in charts, tables, and discussion, the 27 cottons are coded by number according to the increase in the perimeter of the primary wall (Tables I and II).

Fifteen additional cottons were studied to ascertain the effect of processing upon D. Eight of these cottons were selected from the 1950 spinning test cottons of the Section of Cotton and Other Fiber Crops [9]. The other seven constituted a special spinning test conducted for another purpose, but which lent itself to further study in connection with the present investigations. As the particular varieties comprising this series have no bearing on the study, for ease in the presentation of the data the cottons are coded by numbers from 1 to 15. Arealometer A and D values were obtained for the raw stock, card sliver, first and second drawing slivers, and roving of each of the 15 cottons, and for the picker lap of 11. There were in all 86 samples and 3 determinations of A and of D were made for each.

TABLE II. Perimeters as Calculated by Three Different Methods, and Immaturity Ratios

Cotton code number	Primary-wall perimeter[1] (μ)	Perimeter calculated from specific surface A and: Arealometer D^2 (μ)	Perimeter calculated from specific surface A and: Array weight per inch[3] (μ)	Immaturity ratio calculated from arealometer D^2
1	54.4*	36.5	37.1	1.6
2	56.6*	36.0	35.2	1.5
3	61.3	38.6	40.3	1.4
4	65.6	(41.5)	—	(1.7)
5	66.0	42.0	39.5	1.4
6	66.0	(49.7)	—	(1.9)
7	67.2	40.9	42.2†	1.5
8	67.3	43.0	—	1.7
9	68.5	47.7	—	2.1
10	70.0	47.1	45.6†	1.4
11	70.0	(44.9)	(44.5)	(2.0)
12	72.1	46.1	—	1.8
13	72.5*	46.1	—	1.6
14	73.3	47.6	44.5	2.1
15	73.4	47.0	44.9	1.9
16	75.1	45.2	48.4 55.9†	1.5
17	75.2	52.0	47.4	1.9
18	75.2	49.1	48.2	1.8
19	76.1	50.1	46.6	1.7
20	76.5	44.6	45.5†	1.4
21	77.1	52.9	—	2.1
22	79.2	51.4	52.4	2.0
23	79.4	52.7	49.8	1.8
24	80.3	51.9	49.9	1.7
25	88.3	—	—	—
26	89.3	58.0	57.7	2.0
27	90.6	55.6	57.6	2.2

[1] Values are based on 500 fiber measurements except those indicated by *; mean for No. 1 is based on 225 measurements and means for No. 2 and 13 on 250 measurements. For 24 values based on 500 measurements, the least difference significant at the 1% level = 3.19 μ.

[2] Values are the means for seed positions II and III, except those in parentheses, which are for position III only.

[3] Values are the means for positions II and III except the one in parentheses, which is for position III only, and those indicated by †, which are for the total amount of lint. See text page 128 for explanation and for information concerning the two values for No. 16.

Methods of Determining Perimeter

Measurement of the Primary-Wall Perimeter

The procedure for measuring perimeter in the primary-wall stage is described in [7]. Briefly, tufts of fibers cut from young, preserved seeds were fanned out on microscope slides, the fibers stained and allowed to dry adhered to the slides. The ribbon width of 25 fibers in each tuft was measured at the 45% point of tuft length. Twice the width of the flattened fiber is the circumference or fiber perimeter.

For all but 3 of the cottons, 2 fiber tufts were cut from each of 10 seeds, 1 from the chalazal end of the seed and 1 from the middle. Two perimeter values based on 250 observations (25 fibers × 10 seeds) were thus obtained for each cotton. As variance analysis of the tuft means showed no statistically significant difference in the perimeter of fibers from the two different seed positions [7] (Table III), the 2 perimeter values were averaged to give for each cotton 1 value, based on 500 observations.

Unfortunate circumstances caused data to be obtained for only 5 seeds each of Karnak (No. 2) and Off-type Sealand (No. 13) and for only 9 seeds of S × P (No. 1). Moreover, the S × P seeds were so small that only 1 tuft could be cut from each. Thus, the perimeter values for Karnak and Off-type Sealand are based on 250 fiber measurements, that for S × P on 225. An analysis of 92 paired observations (each observation being the mean of 250 measurements) gave, however, the average standard error of a single observation to be 1.44 μ with an average coefficient of variability of 1.91%. These values are small enough to warrant considering, for analytical purposes, the perimeter values for these 3 cottons as comparable with those based on 500 observations.

No new measurements were made for the 9 cottons comprising the earlier study [7].

Calculation of Perimeter from Specific Surface and D

The second set of perimeters was calculated from specific surface A and D values according to the formula of Hertel and Craven [3]. These calculated perimeters were obtained for only 26 cottons, there being no seed cotton available for Rowden 40-5-3-1-2 grown at Shafter, Calif., in 1949.

Since the study was to be concerned in part with the effect of variations in D on perimeter values and since D is related to the flatness (or immaturity) of the fibers [3], it was desirable to have for each of several different perimeters a series of samples that differed in maturity. An attempt was made to obtain the desired samples by hand ginning 100–250 seeds of each cotton and removing separately the fibers from the chalazal (I), midchalazal (II), midmicropylar (III), and micropylar (IV) areas [7]. Since the perimeter of each cotton is assumed to be a definite genetic characteristic and since the degree of maturity has been shown to increase from the chalazal to the micropylar end of the seed [4, 6, 7], the series of 4 samples for each cotton represented 4 different degrees of maturity for that particular perimeter.

The seeds of the Sea Island selection, however, were so small and the fiber population at the micropylar end so sparse that its seeds could be divided for hand-ginning purposes into only 3 areas. The micropylar area was considered lacking in the instances where averages were made.

In all, there were 103 samples (25 cottons × 4 positions + 3 for S × P). Four A and four D determinations were made for all but 13 of these. Nine chalazal and three mid-chalazal samples were too immature to give A readings, and the micropylar sample of S × P was so small that only 2 subsamples could be made from it. The A and D values for the sample of each position were averaged, and from these averages were calculated immaturity ratio (I) and perimeter [3]. For each of the 23 cottons possessing both a No. II and a No. III sample, the perimeter and immaturity ratios for these 2 seed positions were each averaged to give values to represent the cotton as a whole.

It was necessary to obtain A and D for 9 cottons previously studied [7] as the earlier arealometer data * consisted of low-compression values only.

The determination of perimeter from arealometer data will hereafter be referred to as the "D method."

Calculation of Perimeter from Specific Surface and Array Weight per Inch †

The third method of determining perimeter was carried out for only 19 cottons. The calculations were made according to the formula used in the previous perimeter study [4, 7].

Old notation for specific surface:
Array weight per inch × specific surface × 3.942;
or
New notation for specific surface:
Array weight per inch × specific surface × 0.0251.

For 15 of the 19 cottons weight-per-inch values were obtained for seed positions II and III. A perimeter value was calculated for each position, and the cotton as a whole was represented by the average of the two. There was also 1 cotton (No. 11) for which data were obtained for position III only. Of this total of 16 cottons, 9 were those that had been studied earlier [7] and for which weight-per-inch data were already available.

For 3 of the cottons (No. 7, 10, and 20) grown in Shafter, Calif., weight per inch using machine-ginned

TABLE III. Variance Analysis of the Primary-Wall Perimeters of Fibers of 480 Samples Representing the Chalazal End and the Middle of 10 Seeds from Each of 24 Cottons *

Source of variance	Degrees of freedom	Mean square
Cotton	23	12.93†
Position	1	0.41
Cotton × Position	23	0.14
Error	432	0.17‡
Total	479	0.78

* One sample = mean of 25 fiber measurements. Analysis was made on the original observations in eyepiece micrometer units. To convert to perimeter in microns: micrometer units ×2×4.7619.

† Significant at the 1% level.

‡ Standard error of a single observation = 3.90 μ. Standard error of a mean of 20 observations = 0.87 μ. Mean of 480 samples = 74.31 μ. C.V. = 5.24%.

* The earlier measurements of specific surface were made at the University of Tennessee Fiber Research Laboratory, Knoxville, Tennessee.

† Weight-per-inch determinations were made by the Cotton Division, Agricultural Marketing Service, U. S. Department of Agriculture.

lint had been obtained earlier in connection with a related line of investigation. Arealometer data that could be considered comparable with these weight-per-inch values were computed for each of these 3 cottons by weighting the A and D values for the 4 seed positions according to the percentage of lint by weight in the sample in relation to the total lint of the 4 samples. The perimeters calculated for these 3 cottons thus represent the total amount of lint ginned from the seeds. These perimeters, however, fell in line with those based on the average of data for seed positions II and III and are therefore consired comparable to them.

Stoneville 2B (No. 16), grown at Shafter, requires a special explanation. The weight per inch, based on machine-ginned lint, reported for this cotton was 5.03 mg./in., a value very definitely not in agreement with any of the other data. The author considered the discrepancy too large to be accounted for by chance variation in the material or by experimental error and felt justified in assuming that some error occurred either in the labeling of samples or in making records. When this inconsistency became evident, weight-per-inch determinations were then made for the hand-ginned No. II and III samples of the seed cotton on hand that was labeled Stoneville 2B from Shafter. These values and the perimeters calculated from them fell in line with the other data and are the ones used in the analysis. However, in charts and tables, the inconsistent perimeter value is given along with the consistent one.

This third method of determining perimeter will be referred to as the "WPI method."

Comparison of Perimeters Obtained by Three Methods

Before comparing the three sets of perimeter values, it should be pointed out that there is no reason to suppose that the perimeter of the primary wall changes in size as the fiber matures. On drying, however, the mature fiber shrinks roughly from about 32% to 42% [5, 7], with the result that the calculated perimeters for the mature dried fibers are considerably smaller than the primary-wall perimeters. Since it would be logical to assume that differences among cottons in primary-wall perimeters would be paralleled by similar differences in the perimeters of the mature fibers, the primary-wall perimeters are used here as a basis for judging the accuracy of the values obtained by the D method and WPI method. In using the primary-wall perimeters in this way, it is not felt that these data are of absolutely unquestionable accuracy. They are subject to the errors that accompany all such types of measurements and may, in some cases, fail to give a truly representative value for the cotton. Nevertheless, variance analysis of the primary-wall data for 24 cottons gave the standard error of a mean of 20 observations to be 0.87 μ and the C.V. to be 5.24% (Tables II and III). The very small size of these two values would indicate a high degree of consistency in this set of data and justify its use as a standard for judging the reliability of the other methods.

The accuracy of the D-value perimeters may be further checked by comparing them with the WPI perimeters. Since WPI is an actual fiber measurement, while D is a value more or less theoretical in nature, the WPI perimeters might be expected to be more accurate than those calculated with D.

For making over-all comparisons among the three methods, perimeter values representing the cottons as a whole were used. For each cotton the primary-wall perimeter is the average for fibers from the chalazal and middle of the seed areas; the D value and WPI perimeters are the averages for seed positions II and III. As the young seeds supplying the fibers for making primary-wall measurements were much smaller than the mature seeds, it is very probable that the middle portion of the young seeds would correspond to positions II and III of mature seeds. Moreover, it is very likely that the position called chalazal for the young seeds would correspond in many cases to the II position of mature seeds. It was thus assumed that for each type of measurement the averages used represented the bulk of the fiber on a seed and were therefore comparable.

The primary-wall perimeters ranged, for the 27 cottons, from 54.4 μ for No. 1 to 90.6 μ for No. 27 (Table II). Number 2 was slightly coarser than No. 1, while No. 25 and 26 (both Rowden 40-5-3-1-2) were only slightly finer than No. 27. The other 22 cottons formed a series with increasing coarseness from No. 2 to No. 25, except that there was a rather wide gap between No. 24 and the three coarsest cottons.

The primary-wall perimeter data for 24 cottons lent themselves to simple variance analysis. Highly significant differences were found between cottons but not between seed positions or between seed posi-

tions within cottons (Table III). Cotton means based on 20 observations (each observation = mean of 25 fiber measurements) required a difference of 3.19 μ for significance at the 1% level.

An item of interest in this series of measurements is the similarity in perimeter for a variety grown at two different locations, as shown by No. 6 and 8, No. 16 and 18, No. 19 and 20, and No. 25 and 26 (Table II). This similarity gives further support to the belief that fiber perimeter is a genetic character little influenced by environment [4, 7]. The perimeter of Sealand 542, however, which was grown at three locations (No. 4, 9, and 11), varied considerably (Table II).

Perimeters calculated from D ranged from 36.0 μ for No. 2, with No. 1 being only slightly coarser, to 58.0 μ for No. 26, with No. 27 being slightly finer (Table II). Thus the two coarsest and the two finest are the same as for the primary-wall perimeters, though the order of increase in size is reversed. The remaining 22 cottons were distributed between the two extremes, except for the rather wide gap between No. 24 and No. 26 and 27, similar to the gap shown by the primary-wall perimeters. Differences in place of growth resulted for each of the 3 varieties grown at two locations, in rather dissimilar perimeters. It will be shown later that this lack of similarity may possibly be attributed, in part at least, to differences in the maturity of the cottons when grown under different environmental conditions.

The differences among the cottons in *D*-value perimeters were very similar to differences among them in primary-wall perimeters (Figure 1). The correlation coefficient representing the degree of relationship between the two sets of measurements was highly significant from a statistical point of view ($r = 0.935$ for 23 cottons) (Table IV).

Although the correlation between *D* value and primary-wall perimeters was highly significant (Table IV), it was not perfect and therefore some consideration should be given to factors that might possibly be responsible for inconsistencies besides those due to experimental error. One such factor is perhaps the apparently rather large natural variation in *D*. Analyses of the arealometer data for cottons comprising the "Annual Varietal and Environmental Study of Fiber and Spinning Properties of Cotton" [9, 10, 11] and of 89 samples of the present study have shown that the coefficients of variation for *A* values are rather small, while those

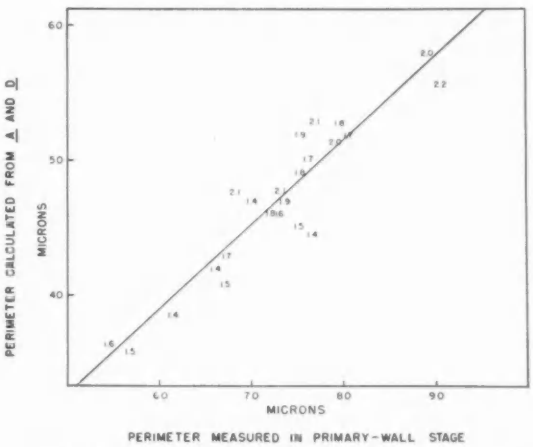

Fig. 1. Regression of perimeters calculated from arealometer specific surface and *D* on primary-wall perimeters for 23 cottons. The plotted points are indicated by the immaturity ratios. Arealometer values are the averages for seed positions II and III. Regression equation: $E = 0.6228X + 1.6484$; $S.E. = 2.10\,\mu$; $r = 0.935$.

TABLE IV. Simple Correlation Coefficients Representing the Degree of Relationship between Perimeters *

Relationship between†		Simple correlation coefficients for seed positions‡				
x	*y*	I	II	III	IV	Av. of II and III
P.W.P. and D.P.		0.951 (17)	0.923 (23)	0.883 (26)	0.841 (26)	0.935 (23)
P.W.P. and D.P.		0.951 (17)	0.950 (17)	0.916 (17)	0.911 (17)	0.943 (17)
P.W.P. and D.P.		—	—	—	—	0.948 (18)
P.W.P. and WPI.P.		—	0.958 (15)	0.966 (15)	—	—
P.W.P. and WPI.P.		—	—	—	—	0.967 (18)§
WPI.P. and D.P.		—	—	—	—	0.942 (18)§
WPI.P. and D.P.		—	0.942 (15)	0.912 (15)	—	0.942 (15)

* Figures in parentheses are the number of paired observations or the number of cottons included.

† Abbreviations: P.W.P.—primary-wall perimeter; D.P.—perimeter calculated from specific surface and *D*; WPI.P.—perimeter calculated from specific surface and array weight per inch.

‡ All correlation coefficients are highly significant.

§ The WPI perimeters for 3 of the cottons represent the total amount of lint.

for D are 4 to 5 times as large.* A second contributing factor may be the fiber profile [7]. Since specific surface values are affected by perimeter variations along the entire length of the fiber and since the primary-wall values are for the 45% point of tuft length, differences among cottons in fiber profile might account for a certain amount of the lack of agreement between the two sets of perimeter values.

There is another factor which deserves perhaps a little more detailed consideration, and that is the maturity of the cotton. The possible importance of maturity can be seen if, in the scatter diagram, the plotted points are indicated by the corresponding immaturity ratios (Figure 1 and Table II). Although the data are not entirely consistent, there is a general tendency for cottons whose calculated perimeters are larger than their primary-wall perimeters would indicate to have high I values (are immature), while others, whose calculated perimeters are smaller than would be expected, tend to have low I values (are mature) (Figure 1). Moreover, for a group of cottons with similar primary-wall perimeters, an increase in calculated values tended to be accompanied by an increase in I. This latter behavior is particularly well illustrated by Stoneville 2B and Deltapine 15 grown at Raleigh, N. C., and Shafter, Calif. All four cottons had practically the same primary-wall perimeter (75.1–76.5 μ; Table II). The Shafter cottons (No. 16 and 20) were more mature and had smaller D-value perimeters than the Raleigh cottons (No. 18 and 19) (Table II; Figure 1).

The possible effect of maturity is further shown by the D-value perimeters for samples representing seed positions, I, II, III, and IV. For the majority of the cottons there was a rather distinct tendency for the perimeter to decrease from chalazal (I) to the micropylar (IV) end of the seed (Table V). Since immaturity ratio decreases in this same direction (Table VI), there would appear to be a tendency for perimeter to decrease as wall thickness increases. Some cottons, however, showed no distinct trend at all (No. 26 and 27; Table V). Moreover, the extent to which the perimeter changed from position to position varied among the cottons.

* Coefficients of variability obtained by Dr. G. T. Den Hartog, Field Crops Research Branch, are as follows: For a set of 88 varieties, 3.3% for A and 13.5% for D; for a second set of 148 varieties, 2.0% for A and 11.7% for D. For 89 samples of the present study, the coefficient of variability for A was 2.8%; that for D was 15.4%.

TABLE V. Perimeters Calculated from Specific Surface and *D*

Cotton code number	Perimeters calculated from specific surface and D for seed positions*			
	I (μ)	II (μ)	III (μ)	IV (μ)
1	38.5	37.2	35.8	33.6
2	37.3	36.4	35.7	34.1
5	41.2	41.0	42.9	38.8
7	43.2	41.5	40.3	35.7
10	47.5	46.5	47.7	41.8
12	49.8	47.1	45.1	46.4
13	47.6	46.9	45.2	44.7
16	50.7	46.9	43.5	42.9
17	51.2	50.6	53.5	53.2
18	49.9	49.7	48.4	49.7
19	48.9	49.8	50.3	50.7
20	47.3	45.1	44.1	44.0
22	51.0	52.8	50.1	47.5
23	52.8	51.4	54.0	51.1
24	55.0	53.1	50.8	48.6
26	57.7	56.9	59.1	58.1
27	55.5	54.7	56.4	55.5

* Values are based on 4 observations each, except for position IV of No. 1, which is based on only 2 observations.

TABLE VI. Immaturity Ratios Calculated from Arealometer *D*

Cotton code number	Seed position*			
	Chalazal I	Mid-chalazal II	Mid-micropylar III	Micropylar IV
1	2.00	1.72	1.53	1.36
2	1.74	1.53	1.46	1.28
3†	—	1.53	1.30	—
4	—	—	1.70	1.30
5	1.59	1.41	1.33	1.13
6	—	—	1.88	1.59
7	1.98	1.59	1.38	1.16
8	—	2.07	1.38	1.16
9	—	2.44	1.80	1.59
10	1.59	1.43	1.36	1.13
11	—	—	1.98	1.72
12	2.58	2.07	1.52	1.50
13	2.09	1.72	1.41	1.36
14	—	2.26	1.88	1.70
15	—	2.10	1.66	1.55
16	2.34	1.72	1.36	1.25
17	2.28	1.93	1.80	1.57
18	2.44	2.05	1.62	1.53
19	2.31	1.89	1.55	1.46
20	1.80	1.48	1.28	1.19
21	—	2.20	2.04	1.66
22	2.68	2.31	1.64	1.36
23	2.22	1.91	1.59	1.41
24	1.80	1.80	1.50	1.33
26	2.60	2.36	1.72	1.93
27	2.33	2.50	1.95	1.68

* Samples too immature to give a specific surface reading are indicated by —.

† Because of their small size, the seeds could be divided into only three areas for hand-ginning purposes.

Thus, in relating the *D*-value perimeters of the different seed positions to the primary-wall perimeters, which are assumed to be constant for the entire seed, inconsistencies in relationships occurred. For example (Figure 2), No. 7 and 17 fall close to the regression line for position I, but at some distance for position IV; and No. 16 and 17 with similar primary-wall perimeters both fall close to the regression line for position I; but, at position IV, No. 17 has a much larger *D*-value perimeter than would be expected, while No. 16 has a much smaller one.

There is, perhaps, some reasonableness in a tendency for perimeter to increase with an increase in immaturity. It is reasonable to assume that, in the undried state, there would be no tendency for perimeter to vary with wall thickness. Since the thin-walled fibers of a particular cotton would have less material to shrink on drying than would thick-walled fibers, it might be expected that, in the dried state, the perimeters of thin-walled fibers would be larger than those of the thick-walled. However, the fact that a few cottons did not show an increase in perimeter with an increase in immaturity would indicate the effect of some factor or factors upon the relative size of *D* other than or in addition to the degree of flatness of the fiber.

The possible effect of maturity is shown in still another way. The scatter diagrams for the 4 seed positions showed an increase in scatter from positions I to IV, and there is consequently a decrease in the size of the correlation coefficient representing the degree of relationship between *D*-value and primary-wall perimeters (Figure 2 and Table IV). Since fiber maturity increases from the chalazal to the micropylar end of the seed (Table VI), it would appear that the more mature a cotton is the less reliable will be its *D*-value perimeters. However, a few cottons were not represented by data for positions I and II and, since some of these were distinctly out of alignment for positions III and IV or both, as for example No. 6 and 21 (Figure 2), it is possible that the high degree of correlation for position I and II may be attributed in part at least to the absence of these particular cottons. When the analysis was based on the 17 cottons that were common to all 4 positions, the correlation coefficients still showed a decrease in size from the chalazal to the micropylar end though the trend was not so

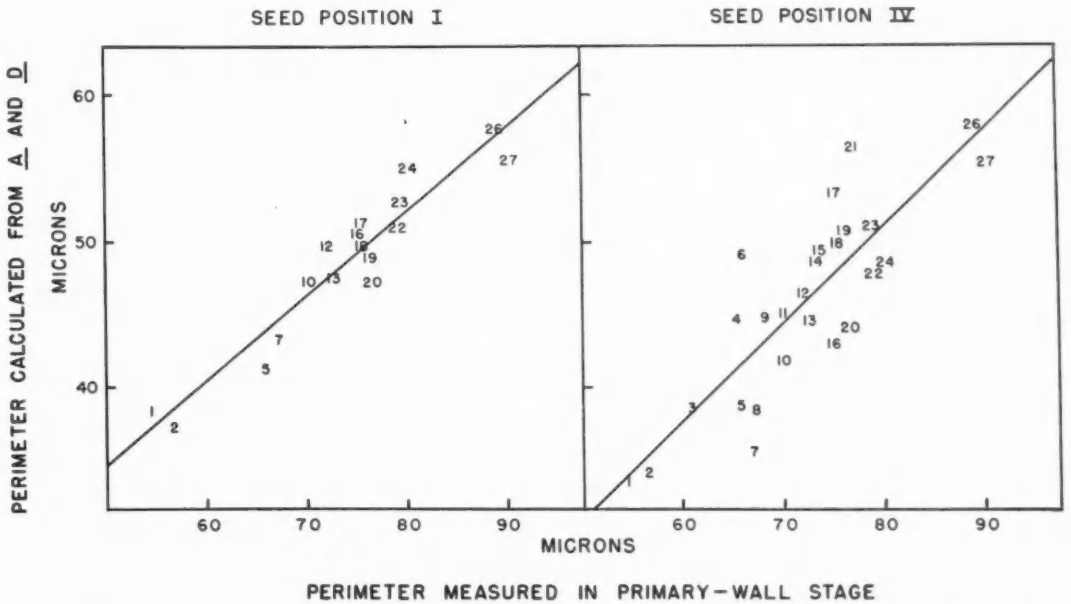

Fig. 2. Regression of perimeters calculated from arealometer specific surface and *D* on primary-wall perimeters for seed positions I (chalazal end of seed) and IV (micropylar end of seed). Plotted points are indicated by cotton code number: I—17 cottons; regression equation, $E = 0.5751X + 6.0557$; $S.E.E. = 1.84\,\mu$; $r = 0.951$. IV—26 cottons; regression equation, $E = 0.6642X - 2.1616$; $S.E.E. = 3.65\,\mu$; $r = 0.841$.

for D are 4 to 5 times as large.* A second contributing factor may be the fiber profile [7]. Since specific surface values are affected by perimeter variations along the entire length of the fiber and since the primary-wall values are for the 45% point of tuft length, differences among cottons in fiber profile might account for a certain amount of the lack of agreement between the two sets of perimeter values.

There is another factor which deserves perhaps a little more detailed consideration, and that is the maturity of the cotton. The possible importance of maturity can be seen if, in the scatter diagram, the plotted points are indicated by the corresponding immaturity ratios (Figure 1 and Table II). Although the data are not entirely consistent, there is a general tendency for cottons whose calculated perimeters are larger than their primary-wall perimeters would indicate to have high I values (are immature), while others, whose calculated perimeters are smaller than would be expected, tend to have low I values (are mature) (Figure 1). Moreover, for a group of cottons with similar primary-wall perimeters, an increase in calculated values tended to be accompanied by an increase in I. This latter behavior is particularly well illustrated by Stoneville 2B and Deltapine 15 grown at Raleigh, N. C., and Shafter, Calif. All four cottons had practically the same primary-wall perimeter (75.1–76.5 μ; Table II). The Shafter cottons (No. 16 and 20) were more mature and had smaller D-value perimeters than the Raleigh cottons (No. 18 and 19) (Table II; Figure 1).

The possible effect of maturity is further shown by the D-value perimeters for samples representing seed positions, I, II, III, and IV. For the majority of the cottons there was a rather distinct tendency for the perimeter to decrease from chalazal (I) to the micropylar (IV) end of the seed (Table V). Since immaturity ratio decreases in this same direction (Table VI), there would appear to be a tendency for perimeter to decrease as wall thickness increases. Some cottons, however, showed no distinct trend at all (No. 26 and 27; Table V). Moreover, the extent to which the perimeter changed from position to position varied among the cottons.

* Coefficients of variability obtained by Dr. G. T. Den Hartog, Field Crops Research Branch, are as follows: For a set of 88 varieties, 3.3% for A and 13.5% for D; for a second set of 148 varieties, 2.0% for A and 11.7% for D. For 89 samples of the present study, the coefficient of variability for A was 2.8%; that for D was 15.4%.

TABLE V. Perimeters Calculated from Specific Surface and *D*

Cotton code number	Perimeters calculated from specific surface and D for seed positions* I (μ)	II (μ)	III (μ)	IV (μ)
1	38.5	37.2	35.8	33.6
2	37.3	36.4	35.7	34.1
5	41.2	41.0	42.9	38.8
7	43.2	41.5	40.3	35.7
10	47.5	46.5	47.7	41.8
12	49.8	47.1	45.1	46.4
13	47.6	46.9	45.2	44.7
16	50.7	46.9	43.5	42.9
17	51.2	50.6	53.5	53.2
18	49.9	49.7	48.4	49.7
19	48.9	49.8	50.3	50.7
20	47.3	45.1	44.1	44.0
22	51.0	52.8	50.1	47.5
23	52.8	51.4	54.0	51.1
24	55.0	53.1	50.8	48.6
26	57.7	56.9	59.1	58.1
27	55.5	54.7	56.4	55.5

* Values are based on 4 observations each, except for position IV of No. 1, which is based on only 2 observations.

TABLE VI. Immaturity Ratios Calculated from Arealometer *D*

Cotton code number	Seed position* Chalazal I	Mid-chalazal II	Mid-micropylar III	Micropylar IV
1	2.00	1.72	1.53	1.36
2	1.74	1.53	1.46	1.28
3†	—	1.53	1.30	—
4	—	—	1.70	1.30
5	1.59	1.41	1.33	1.13
6	—	—	1.88	1.59
7	1.98	1.59	1.38	1.16
8	—	2.07	1.38	1.16
9	—	2.44	1.80	1.59
10	1.59	1.43	1.36	1.13
11	—	—	1.98	1.72
12	2.58	2.07	1.52	1.50
13	2.09	1.72	1.41	1.36
14	—	2.26	1.88	1.70
15	—	2.10	1.66	1.55
16	2.34	1.72	1.36	1.25
17	2.28	1.93	1.80	1.57
18	2.44	2.05	1.62	1.53
19	2.31	1.89	1.55	1.46
20	1.80	1.48	1.28	1.19
21	—	2.20	2.04	1.66
22	2.68	2.31	1.64	1.36
23	2.22	1.91	1.59	1.41
24	1.80	1.80	1.50	1.33
26	2.60	2.36	1.72	1.93
27	2.33	2.50	1.95	1.68

* Samples too immature to give a specific surface reading are indicated by —.

† Because of their small size, the seeds could be divided into only three areas for hand-ginning purposes.

Thus, in relating the D-value perimeters of the different seed positions to the primary-wall perimeters, which are assumed to be constant for the entire seed, inconsistencies in relationships occurred. For example (Figure 2), No. 7 and 17 fall close to the regression line for position I, but at some distance for position IV; and No. 16 and 17 with similar primary-wall perimeters both fall close to the regression line for position I; but, at position IV, No. 17 has a much larger D-value perimeter than would be expected, while No. 16 has a much smaller one.

There is, perhaps, some reasonableness in a tendency for perimeter to increase with an increase in immaturity. It is reasonable to assume that, in the undried state, there would be no tendency for perimeter to vary with wall thickness. Since the thin-walled fibers of a particular cotton would have less material to shrink on drying than would thick-walled fibers, it might be expected that, in the dried state, the perimeters of thin-walled fibers would be larger than those of the thick-walled. However, the fact that a few cottons did not show an increase in perimeter with an increase in immaturity would indicate the effect of some factor or factors upon the relative size of D other than or in addition to the degree of flatness of the fiber.

The possible effect of maturity is shown in still another way. The scatter diagrams for the 4 seed positions showed an increase in scatter from positions I to IV, and there is consequently a decrease in the size of the correlation coefficient representing the degree of relationship between D-value and primary-wall perimeters (Figure 2 and Table IV). Since fiber maturity increases from the chalazal to the micropylar end of the seed (Table VI), it would appear that the more mature a cotton is the less reliable will be its D-value perimeters. However, a few cottons were not represented by data for positions I and II and, since some of these were distinctly out of alignment for positions III and IV or both, as for example No. 6 and 21 (Figure 2), it is possible that the high degree of correlation for position I and II may be attributed in part at least to the absence of these particular cottons. When the analysis was based on the 17 cottons that were common to all 4 positions, the correlation coefficients still showed a decrease in size from the chalazal to the micropylar end though the trend was not so

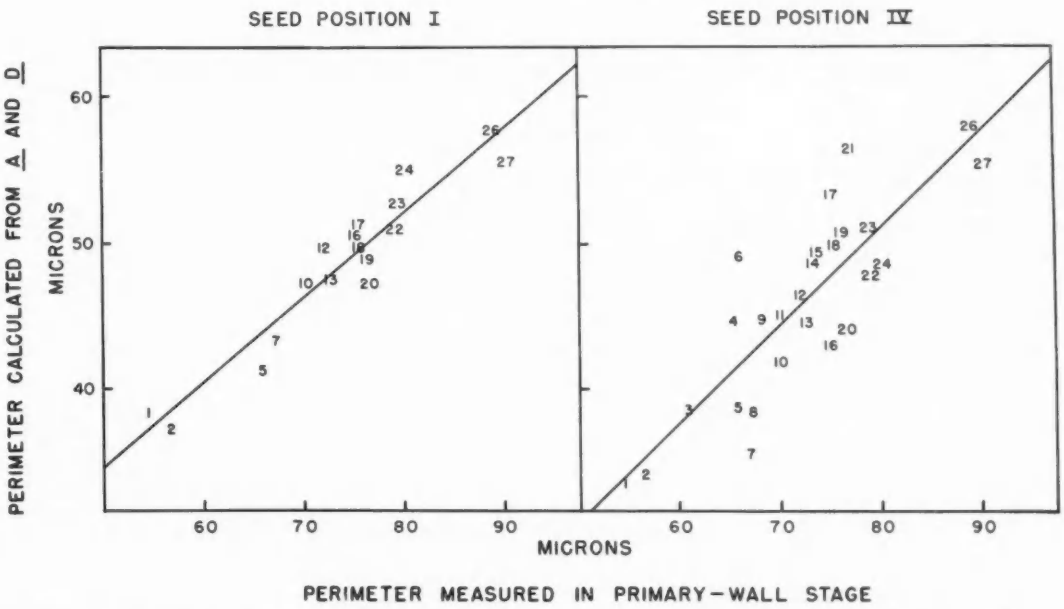

Fig. 2. Regression of perimeters calculated from arealometer specific surface and D on primary-wall perimeters for seed positions I (chalazal end of seed) and IV (micropylar end of seed). Plotted points are indicated by cotton code number: I—17 cottons; regression equation, $E = 0.5751X + 6.0557$; $S.E.E. = 1.84\,\mu$; $r = 0.951$. IV—26 cottons; regression equation, $E = 0.6642X - 2.1616$; $S.E.E. = 3.65\,\mu$; $r = 0.841$.

striking as with the complete data (Table IV). Consequently, the possibility of a decrease in the reliability of D-value perimeters with an increase in maturity cannot be completely disregarded.

The maturity relationships are not at all clear, and this phase of the problem is being investigated further.

Perimeters calculated from WPI were obtained for only 19 of the 27 cottons, but among them were those shown by the other two methods to be the 2 finest and the 2 coarsest. As with the method using D, No. 2 was the finest, with No. 1 being slightly coarser, while No. 26 was the coarsest, with No. 27 being slightly finer (Table II). The remaining 13 cottons were distributed between the two extremes. The gap between the 2 coarsest cottons and the rest of the group shown by the other 2 sets of perimeter values is shown also by this one. As this gap was shown by all 3 sets of perimeter measurements the question naturally arises as to whether it represents a similar gap for the American Upland cottons taken as a whole. Similarity in perimeter size with different locations of growth was shown most strikingly by both No. 16 and 18, and No. 19 and 20 (Table II).

Differences among cottons in the WPI perimeters were very similar to the differences among the same cottons in the primary-wall perimeters, the correlation coefficient representing the degree of relationship between the two sets of values being highly significant (Table IV). If the correlation coefficients are based on the same cottons (18; Table IV), differences in WPI perimeters appeared to follow differences in primary-wall perimeters to a slightly more precise degree than the D-value perimeters ($r = 0.967$ and 0.948, respectively).

The degree of relationship between the *two sets of calculated perimeters* is best shown by a scatter diagram in which the line for the regression of D-value perimeters on WPI perimeters may be compared with the line representing complete agreement between the two sets of measurements. As can be seen from Figure 3, the regression line for the actual data nearly coincides with that of complete agreement. The correlation coefficient representing the degree of relationship between these sets of calculated perimeters is highly significant ($r = 0.942$) (Table IV). Inconsistencies do not appear to be related to maturity differences among the cottons nor would it be expected that they could be explained in part by differences in fiber profile since weight per inch as well as specific surface would take into consideration variations in profile along the length of the fiber. A certain amount of the lack of agreement may possibly be attributed to the rather large variability of D.

From these comparisons it may be concluded that on the whole for unprocessed samples perimeters calculated from arealometer values will show differences between cottons that parallel those obtained by measurements in the primary-wall stage and will differ little from those calculated from specific surface and array weight per inch.

Further evidence of the accuracy of the values calculated from A and D is shown by the extent to which calculated weight per inch agrees with array weight per inch. There were 31 samples for which both types of weight-per-inch data were obtained; 15 samples for seed position II and 16 for position III. The line representing the regression of arealometer weight per inch on array weight per inch differed very little from that of complete agreement between the two sets of measurements. The correlation coef-

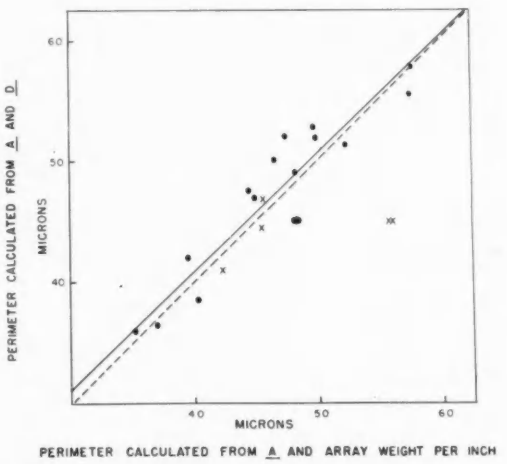

Fig. 3. Regression for 18 cottons of perimeters calculated from specific surface and D on perimeters calculated from specific surface and array weight per inch. Perimeters indicated are the average for seed positions II and III; those indicated by × represent the total amount of lint. Points indicated by doubled signs represent a consistent and inconsistent weight-per-inch value for Stoneville 2B (No. 16) grown at Shafter, Calif., the inconsistent perimeter value not being included in the calculations (see page 128 for explanation). Regression equation: $E = 0.9737X + 1.9001$; $S.E. = 2.26\,\mu$; $r = 0.942$.

ficient representing the degree of relationship between the two was 0.970, a highly significant value.

Variations in *D* with Processing

It was stated in the Introduction that the *D* value of second drawing sliver was observed to be almost invariably larger than that of the ginned lint of the same cotton [9, 10, 11]. This increase in *D* can not conceivably be attributed to an actual increase in immaturity during the process of converting raw stock into second drawing sliver. It has been suggested, however, that the breaking off of the very slender tips of the fibers during the various processing stages and their subsequent loss from the lint could account for some, if not all, of this increase in *D;* the tip is assumed to be more mature than the rest of the fiber, for its walls would be thicker in relation to the size of the lumen than they would be at other portions along the fiber length; the loss of these mature tips would tend to decrease the general maturity of the cotton and thus bring about an increase in *D*.* Although the manipulation the cotton receives during the various processing stages undoubtedly results in some tip breakage, it does not seem reasonable to suppose that under most conditions there would be enough tip loss to affect *D* appreciably. Moreover, a very superficial examination of fibers from the second drawing sliver of several cottons did not reveal any excessive amount of tip loss.

It was therefore considered desirable to obtain more extensive information regarding the effect of processing upon the *D* value of the raw stock, in order to ascertain the extent to which the kind of material selected for making arealometer determinations might affect the reliability of these data and the values calculated from them.

With but few exceptions among the spinning products of the 15 cottons studied, the *D* value was observed to decrease from raw stock † to picker lap, and then to increase with each of the 4 following products: card sliver, first and second drawing slivers, and roving. On the whole, the carding action nearly compensated for the decrease in *D* that was brought about by the picker, for the *D* values of card sliver were about the same as those for the ginned lint. The greatest increase in *D* was from the second drawing sliver to roving, though the increase from card sliver to first drawing sliver was only slightly less. The most pronounced increase in *D*

* These suggestions were made orally by Dr. K. L. Hertel and Dr. C. J. Craven of the University of Tennessee.

† "Raw stock" for these cottons is not exactly synonymous with "ginned lint" for a few of the cottons had been packaged in small bales. The effect of baling on the *D* value cannot be totally disregarded, yet there was observed to be no consistent relationship between the extent to which the *D* value was decreased by the picker and the type of packaging for the raw stock.

TABLE VII. Arealometer *D* Values

Cotton code number	Range in specific surface	Arealometer *D* values for*					
		Raw stock	Picker lap	Card sliver	First drawing sliver	Second drawing sliver	Roving
1	402–420	5	—	3	10	17	32
2	422–438	8	—	6	18	24	33
3	424–447	18	12	20	27	35	51
4	429–453	17	15	18	32	32	49
5	442–462	20	15	20	33	38	46
6	444–462	15	7	13	28	33	45
7	452–468	24	18	25	38	48	56
8	465–488	25	24	24	40	42	58
9	477–495	31	27	25	39	46	61
10	491–518	43	27	30	46	42	68
11	502–514	26	22	21	32	48	54
12	511–532	26	—	23	34	42	53
13	524–556	31	—	33	56	51	71
14	537–554	51	40	40	52	59	78
15	559–579	49	35	37	50	59	69
Average change in *D*			−7.0†	+2.8†	+13.1‡	+5.4‡	+13.9‡

* Each value is the mean of 3 observations.
† Based on 11 cottons.
‡ Based on 15 cottons.

would thus appear to take place at the stages where the greatest drafting occurs (Table VII). The A values were altered very little, though there was a slight tendency for specific surface to increase with processing, which trend would indicate a slight increase in immaturity, possibly attributable to tip loss.

As a result of the rather large changes in D and the very slight alterations in A with processing, the calculated values of immaturity ratio and perimeter, etc., will vary for the different materials as the D value varies (Table VIII). Obviously, these differences in the calculated values do not represent actual differences in these fiber properties.

The effect of processing may be further shown by comparing the results obtained at the Knoxville and Clemson laboratories on the same cottons. At the Knoxville laboratory, the materials for securing arealometer data are taken from the samples prepared for making fibrograph readings and thus have received considerable combing. At the Clemson laboratory, the samples for making arealometer determinations were not combed but were gently teased apart with needles until the fiber clumps were separated and a fairly even distribution of the fibers was achieved. The A values for both raw stock and second drawing sliver were very similar for both laboratories (Table IX). Too, there was little difference between the laboratories in the D values for second drawing sliver, but the Knoxville D values for raw stock were much larger than those for Clemson. Apparently the combing the raw stock received in preparing the Knoxville samples was sufficient to decrease considerably the resistance to air flow under high compression and thus resulted in D values larger than those of the uncombed Clemson samples. Combing, however, appeared to have little effect on the resistance offered by materials which had already been subjected to drafting since the Knoxville D values for second drawing sliver differed little from those of Clemson.

Hertel and Craven in the conclusion to their paper on measurements by the means of the arealometer state ([3], pages 773–4) that ". . . the increase in the apparent specific surface as observed on the arealometer at low porosity over that at high porosity is correlated with the immaturity of the cotton." They further state that their suggested explanation for the correlation is ([3], page 774) ". . . probably not strictly true but it is approximately true over the limited range of the cottons tested." It is on the assumption, however, that the variations in D are overwhelmingly a function of immaturity that Hertel and Craven developed the formulas to calculate immaturity ratio, perimeter, weight per inch, and wall thickness.

The results given in this paper for unprocessed cotton support Hertel and Craven's conclusions, since perimeters calculated from A and D differed very little from those calculated from array weight per inch and the calculated weight-per-inch values differed little from those obtained by the array method. On the other hand, the results showed that with processed samples the calculated values would vary with the material being tested. Particularly would this be true of materials that had been subjected to considerable drafting, since D was shown to increase from card sliver through each stage of drafting to roving, while there was little change in A. To explain the observed changes in the value of D, it must be assumed that for processed materials D is not solely a function of immaturity but is also a function of changes that occur during processing, particularly during drafting.

It is outside the limits of this paper to discuss the factors in processing which are responsible for changes in the apparent specific surface at low porosity (high compression). However, since processing does influence the value of D, it follows that the formulas developed by Hertel and Craven for calculating perimeter, etc., from A and D [3] are not valid on processed samples, particularly those that have had considerable drafting. These formulas are, however, apparently valid on ginned lint samples where D is overwhelmingly a function of immaturity.

TABLE VIII. Fiber Perimeters Calculated from Specific Surface and *D*

Cotton code number	Perimeters calculated from specific surface and D for: Raw stock (μ)	Picker lap (μ)	Card sliver (μ)	First drawing sliver (μ)	Second drawing sliver (μ)	Roving (μ)
3	43.6	40.2	44.3	48.7	52.6	60.1
4	43.3	41.6	42.5	49.9	50.3	58.5
5	44.0	40.5	42.7	49.6	53.1	55.9
6	40.5	34.4	38.7	46.8	49.7	55.5
7	44.7	41.8	45.7	52.3	56.1	59.6
8	43.9	44.2	43.3	51.0	52.2	57.9
9	46.9	44.7	43.4	49.8	52.2	59.5
10	48.6	43.5	44.1	50.8	49.8	59.8
11	41.9	39.4	39.0	44.0	52.3	54.2
14	48.9	45.6	44.4	48.9	52.5	58.1
15	45.7	41.8	41.9	47.3	50.8	53.0

Summary

A study was undertaken to investigate the reliability of perimeter values calculated by means of the arealometer. Perimeters for a series of 27 cottons representing a wide range in fineness were obtained by three different methods: (*1*) measurement in the primary-wall stage—27 cottons; (*2*) calculation from arealometer values-specific surface (*A*) and *D*—26 cottons; and (*3*) calculation from *A* and array weight per inch—19 cottons.

Perimeters calculated by the rapid arealometer method showed differences among cottons that paralleled those shown by the primary-wall perimeters. The correlation coefficient representing the degree of relationship between the two sets of values was highly significant. There was a general tendency for cottons whose *D*-value perimeters were larger than their primary-wall perimeters would indicate to be immature, while cottons whose *D*-value perimeters were smaller, tended to be mature. Moreover, the majority of the cottons showed, for a series of samples that were specially prepared for each cotton to represent an increase in immaturity, that perimeter tended to increase as immaturity increased. A few, however, showed a decrease in perimeter with an increase in immaturity.

The *D*-value perimeters were very similar to those calculated from specific surface and array weight per inch. The regression line representing the relationship between the two sets of measurements practically coincide with the line representing complete agreement. Moreover, calculated weight-per-inch values differed little from those obtained by array weight per inch.

For a second set of cottons (15 in number) arealometer determinations were made and perimeters calculated for raw stock, picker lap (11 cottons), card sliver, first and second drawing sliver, and roving. With but few exceptions, the *D* value, and consequently the perimeter, etc., decreased from raw stock to picker lap and increased successively for card sliver, first and second drawing sliver, and roving. The greatest changes in *D* occurred in the conversion of card sliver to first drawing sliver and of second drawing sliver to roving, thus corresponding to the stages where the greatest drafting occurs.

The results indicate that the formulas for calculating immaturity ratio, perimeter, weight per inch, and wall thickness from arealometer values obtained at low and at high compression are valid for ginned lint but are not valid for samples that have been subjected to considerable mechanical drafting.

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TABLE IX. Arealometer Specific Surface and *D* Values Obtained at the Knoxville and Clemson Laboratories *

Cotton code number	Raw stock				Second drawing sliver			
	A		*D*		*A*		*D*	
	Knox.	Clem.	Knox.	Clem.	Knox.	Clem.	Knox.	Clem.
1	409	402	20	5	410	416	25	17
2	430	422	21	8	432	437	26	24
6	459	444	28	15	457	460	35	33
7	452	460	30	24	461	468	41	48
11	500	504	38	26	505	502	43	48
12	505	511	38	26	520	522	48	42
13	559	524	65	31	557	555	60	51
15	569	579	58	49	551	560	58	59

* University of Tennessee Fiber Research Laboratory, Knoxville, Tennessee, and Field Laboratory of the Section of Cotton and Other Fiber Crops, Field Crops Research Branch, U. S. Department of Agriculture.

Brown of the Textile School, Clemson College, Clemson, South Carolina, for the use of the arealometer.

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(Manuscript received September 3, 1954.)

Correction

Fine Structure in Viscose Fibers

G. D. Joshi and J. M. Preston

November 1954 Volume XXIV, No. 11 Page 973

In Figure 3, parts *b* and *d* should have been reversed top for bottom for easier comparison. Also, the plane of polarization for part *a* is NE-SW, and that for part *b* is N-S, instead of the reverse, as originally stated.

Letters to the Editor

Short communications in the form of Letters to the Editor are intended to provide prompt publication of significant new research results and to permit an exchange of views on papers previously published in the JOURNAL. These communications are not submitted to formal review as are research papers, and the editors do not assume any share of the author's responsibility for the information given or the opinions expressed. When work previously published in the JOURNAL is the subject of critical comment, the authors of the original paper are given an opportunity to submit a reply, which will be published concurrently when possible.

Degradation of Cotton during Mechanical Processing

INDIAN CENTRAL COTTON COMMITTEE
Technological Laboratory
Matunga, Bombay, India
September 27, 1954

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

Whitwell and his collaborators [5, 6] observed that there was significant chemical degradation in cotton cellulose during the different stages of mechanical processing in the manufacture of yarn, as shown by the decrease in its molecular weight. For this purpose, they measured the intrinsic viscosity in cupriethylenediamine of samples drawn at different stages from bale cotton to yarn. It was found that this degradation was significant enough to be considered of practical importance. In order to ascertain whether the mechanical processing of Indian cottons produced any chemical degradation, and if so to what extent, work was started at this laboratory with slight modifications in the above method, and considerable data have been obtained. Before the publication of these results, Conrad and Rusca [2] published a paper in which it is shown that no demonstrable chemical degradation occurs during the mechanical processing. Whitwell and Schwenker [3, 7, 8] have, however, substantiated their earlier findings in subsequent papers. As it is considered that some observations made in the present experiments would throw some light on this subject, a brief account is given here, but the details will be published in due course.

In these experiments, four Indian cottons, viz., Laxmi, Suyog, Cambodia Co. 4, and Gaorani 6 were tested. For this purpose lint samples were given a cold wash with a mixture of benzene and alcohol to facilitate quick wetting in the solvent. Two additional sets of preliminary treatments were also experimented upon: (1) a hot alcohol extraction for 4 hr followed by washing in distilled water, and (2) hot alcohol extraction followed by a mild alkali boil in a 0.25% solution of caustic soda for 4 hr, followed by scouring in dilute acetic acid and washing in distilled water. All samples were dried at room temperature. Treated samples of lint were cut with scissors to about ⅛-in. length, and the viscosity of a 0.5% solution of lint in cuprammonium hydroxide was determined by the standard method [4]. The fall in viscosity values of samples drawn at different stages of processing was taken as a relative measure of degradation [1]. The results obtained are summarized in Table I.

It will be seen that the viscosity values of samples extracted both with cold and hot organic solvents are practically the same and have shown a definite fall from raw cotton to yarn.

Further, it will be noticed that a simple mild alkali treatment has brought about an appreciable fall in the viscosity values in comparison with those of the alcohol-treated samples. In the case of cold or hot organic solvent treatments, yarn gives the lowest viscosity for all the samples, but with an alkali boil it is lower in two cottons, higher in one, and same in another, compared with that of the double roller-ginned cotton. The values for scutcher samples are also very erratic. This inconsistency indicates that the earlier trend in the degradation due to mechanical processing from raw cotton to yarn is offset by the subsequent wet processing.

The results obtained in the present experiments, therefore, show that there is a definite measurable chemical degradation due to mechanical processing

TABLE I. Viscosity Values of 0.5% Solution (in Poises)

Nature of Treatment	Laxmi				Suyog				Cambodia Co. 4				Gaorani 6			
	D.R.* Ginned Cotton	Scutcher Lap	Yarn	Fall from D.R. Cotton to Yarn (%)	D.R. Ginned Cotton	Scutcher Lap	Yarn	Fall from D.R. Cotton to Yarn (%)	D.R. Ginned Cotton	Scutcher Lap	Yarn	Fall from D.R. Cotton to Yarn (%)	D.R. Ginned Cotton	Scutcher Lap	Yarn	Fall from D.R. Cotton to Yarn (%)
Cold benzene-alcohol treatment	0.5427	0.4889	0.4945	8.9	0.5142	0.5055	0.4855	5.6	0.6105	0.5344	0.5091	16.6	0.6608	0.6328	0.6184	6.4
Hot alcohol extraction treatment	0.5542	0.4888	0.4593	11.1	0.5259	0.5072	0.4724	10.2	0.5840	0.5562	0.5117	12.4	0.6476	0.6302	0.6016	7.1
Hot alcohol extraction followed by an alkali boil treatment	0.3562	0.3344	0.2593	27.2	0.3395	0.3309	0.3793	−11.7	0.3672	0.3342	0.3671	0.0	0.4526	0.4088	0.4088	9.7

* Double roller.

in the manufacture of yarn, but it is not markedly significant in comparison with the subsequent degradation brought about by wet processing.

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K. S. BHUJANG
C. NANJUNDAYYA

TEXTILE RESEARCH INSTITUTE
Princeton, New Jersey
November 24, 1954

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In a recent article [2], the writers of this letter stated:

"The results of the wet processing data discussed here indicate that cotton samples of different molecular weights before (wet) processing may be degraded to almost the same molecular weight level as a result of this processing. If this is the pattern for all wet processing . . . , then the degradation due to mechanical processing is of little practical importance."

In a Letter to the Editor of TEXTILE RESEARCH JOURNAL published with this letter, Bhujang and Nanjundayya have submitted data on the effect of a "mild wet process" on four Indian cottons and on samples of each which were mechanically processed; the results purport to prove that the above speculation regarding elimination of mechanical degradation effects by chemical treatments is true. It is gratifying to see that their data on the mechanical action

confirm the degradation which we have previously reported.

It is, however, unfortunate that their results do not offer convincing evidence of the effect of the mild wet process (0.25% NaOH for 4 hr). If one does not include the data for the chemical process, their results are quite consistent, but the results of the caustic treatment indicate both increases and decreases in viscosities rather than a constant trend. These results would indicate something erratic in the caustic action; it is not possible to speculate on the cause since the details of the experiment are not provided.

From the data presented we can only conclude that Bhujang and Nanjundayya are not justified in their statement that "the . . . trend in degradation due to mechanical processing from raw cotton to yarn is offset by the subsequent wet processing."

Further, unpublished data [1] have been made available to us which indicate the following. A treatment with 0.4% NaOH for 36 hr at 60°C on two cottons, one of which had been measurably degraded mechanically, eliminated no portion of the difference existing between samples prior to the alkali treatment. Replication of results and samples at various time intervals indicated that the treatment was well controlled.

In the light of the above remarks, we find Bhujang and Nanjundayya's conclusions interesting, not impossible, but certainly not decisive.

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J. C. WHITWELL
R. F. SCHWENKER, JR.

Colloquium

on

Rheological Properties of Dilute Polymer Solutions

A Colloquium on Rheological Properties of Dilute Polymer Solutions will be held at or near Amsterdam, Holland, in July 1955, shortly before the Congress of the International Union of Chemistry in Zurich.

At the Colloquium, a limited number of papers will be read by invited speakers. The following subjects will be discussed: (1) viscoelastic properties; (2) non-Newtonian behavior; (3) anomalous behavior at extreme dilution, including polyelectrolytes; (4) flow birefringence. The official language at the Colloquium will be English; French and German are admitted.

The Netherlands Rheological Society will act as the Organizing Committee. Communications should be sent to the Organizing Committee, c/o Dr. F. Schwarzl, Central Laboratory, T.N.O., Julianalaan 134, Delft, Netherlands.

INDUSTRIAL SECTION

Simplification of Yarn Numbering

Preparation for the Introduction of the International Numbering of Yarns in Germany

Kurt Hentschel[1]

Chief Engineer, German Textile Standardization Committee

Foreword

The accompanying article by Mr. Kurt Hentschel will be of general interest to the United States textile industry, and particularly to those parts of the industry that produce or use yarns made of various fibers. The single universal numbering system for all yarns referred to in the article is intended to displace eventually the confusing multiplicity of systems now used (see ASTM D 861–52). The proposal to recognize a series of preferred yarn numbers and to produce other yarns only on special order aims to bring to the textile industry the well-known benefits of simplified practice.

Mr. Hentschel is Chief Engineer of the German Textile Standardization Committee (Oberingenieur Deutscher Normenausschuss, Subcommittee Textilnorm). He has been active in promoting standardization of practices and machinery in the German textile industry. He was the official delegate from Germany to the meeting of the International Organization for Standardization, Committee ISO/TC/38, in New York in 1952, where he took an active part in the work of drafting standard yarn test methods. He was Germany's unofficial observer at the Bournemouth conference of ISO/TC/38 in 1951. He has written several papers on yarn simplification which have been published in *Textil-Praxis*, a leading German textile publication.

The article was translated from the German manuscript by Mrs. Ilse Zeise and has been edited by Dr. A. G. Scroggie of E. I. du Pont de Nemours, Inc. Dr. Scroggie is chairman of a committee of the American Society for Testing Materials which deals with the international yarn testing methods for the American Standards Association. The latter represents the United States on the Technical Committees on Textiles (TC/38) of the International Organization for Standardization (ISO). In its capacity as a co-ordinating agency, the American Standards Association has a Committee (L-23) for ISO/TC/38, composed of representatives from interested segments of the United States industry. This committee has arranged for the publication of this article.

Comments and requests for further information about the ISO work should be addressed to the American Standards Association, Committee L-23, 70 East 45 Street, New York 17, N. Y.

EDITOR

THE TECHNICAL Committee on Textiles, ISO/TC/38, of the International Organization for Standardization resolved in its meeting in Bournemouth, England, in June 1951, as follows:

Technical Committee on Textiles ISO/TC/38 reaffirms the proposal to use, for all textile materials, one direct, decimal numbering system, based on metric units, namely, grams for weight and meters for length and their multiples or submultiples; the specific units used must be stated in all cases. The combination grams-per-ten-kilometers is designated as a "grex." The combination grams-per-kilometer is designated as a "tex." It is recommended that member countries encourage the use of this system in their own countries.

The question of the numbering of yarns has not been discussed again by the Main Committee ISO/

[1] Translated by Ilse Zeise.

TC/38 since that time, but the Secretariat of the Subcommittee on "Preferred Series for Yarn Numbers" was assigned to the Standards Institute of the Netherlands (Centraal Normalisatie Bureau) in a meeting of ISO/TC/38 at Buxton, England, in 1948; and this Committee has attempted, by two circular letters to the 38 countries participating in the ISO Committee, to further advance the work assigned to it, i.e., to develop a preferred series of numbers for yarn counts in the new numbering system. In the three years which have passed since the resolution was adopted at Bournemouth, various branches of the German textile industry have considered the question of the numbering of yarns very thoroughly. The author of this contribution himself has informed the German textile industry in three articles in *Textil-Praxis* [1] about the previous history of the new numbering system and its details, and has also made some proposals which caused further discussions in the German textile industry. In these considerations the question of which numbering unit (grex, tex, or other multiples of these units [2]) should be chosen plays only a subordinate role. For the predominant part of the German textile industry only the tex unit will be of interest. The chemical (man-made) fiber industry will probably choose the grex unit, since it is closer to the denier unit used heretofore. The selection of a unit will be more difficult for the manufacturers of coarse yarns, for which the author suggested the unit g/10 m with the name mex. The author wishes to thank Jacques Corbiére, who referred in [2] to an error on the author's part in the explanation of the unit, "mex," proposed by him. He agrees to Corbiére's suggestion to define the unit "mex" as 10^4 grex or "myriagrex," equivalent to 1 g/m.

The discussions in the German textile industry have shown that the difficulties confronting the introduction in commerce of the new international system of numbering of yarns are not primarily of a scientific but rather of a practical nature. It is not easy to change from the length per unit weight numbering to the weight per unit length numbering system, when one has thought all one's life in length numbers for yarns. Moreover, it is rather important to examine which individual numbers (or counts) should be used in the new system of weight numbering, because most branches of the German textile industry wish to combine the change to the new system of yarn numbering with a standardization of the production program of the spinning mills on an international basis, if possible. Consequently, two questions must be examined in advance:

1. Is it possible to establish a uniform series of preferred yarn numbers (or counts) for all branches of the textile industry? That was the task which was assigned in Buxton to the Centraal Normalisatie Bureau and the Subcommittee conducted by it.

2. What steps can facilitate accustoming practical men to the change from a length numbering to a weight numbering system?

The investigations which the German textile industry has carried out on the question of a preferred series of yarn numbers have shown clearly that it is not possible to select a series of yarn numbers which can be used uniformly by all branches of the spinning industry. Even in the ranges in which the yarn numbers manufactured by the different branches of the spinning industry overlap, the requirements to be met by the fineness of the graduation, i.e., closeness of the numbers, differ, so that the establishment of a uniform series of yarn numbers for one industry will never meet the requirements of another industry nor give a basis for a sound standardization of the production program of the spinning mills. On the other hand, it would be regrettable if, at the conversion to the international weight numbering, complete freedom would be allowed in the choice of the yarn counts. The consequence would be, as is the case now, that yarns lying close together, and having practically the same number when the unavoidable fluctuations in the actual size made (tolerances) are taken into consideration, would be designated by different numbers. This consideration led to the search for a numerical series useful for yarn numbers, a series which is regularly graded at such a fine scale that, even in the case of extremely uniform yarns, e.g. $\pm 3\%$ fluctuation, sufficient numbers are available for the designation of two adjacent yarns. This requirement would be met by a numerical series with an interval between steps of 6%. The complete series would be sufficient to designate, with a permissible number fluctuation of $\pm 3\%$, all yarns which can be differentiated in practice. A series with an interval between steps of 6% has already been standardized internationally. It is the series R40 of standard figures. For practical use as a yarn number series, it is possible to round off still further without hesitation, in order to obtain values

[2] See ASTM D 861-52 for definitions and further details.

for yarn numbers which can be handled as easily as possible. Table I gives the values for a yarn number series which were obtained by rounding off series R40.

The complete series would not be used by any branch of the spinning industry since, as a rule, larger number fluctuations than ±3% must be expected and consequently a coarser gradation in the numerical series has developed in practice. However, it is just as important that other numbers than those given in Table I, numbers which are not required for the purposes of yarn numbering, should not be used. Each intermediate number, e.g., number 62 between 60 and 64, or number 29 between 28 and 30, would fall within the range of the tolerance field of the two adjacent numbers (±3%) and is therefore superfluous. This fact is of fundamental importance for the conversion of the numbering systems used heretofore to the proposed international weight numbering system and for the standardization of the production programs to be combined therewith, as can be seen also from the following considerations on the second question, i.e., steps to facilitate becoming accustomed to the conversion.

TABLE I. Rounded-Off Standard Figures of Series R40 for the Purposes of Yarn Numbering (Proposal)

Rounded Values for Yarn Numbers *	Rounded Values for Yarn Numbers *
10	32
10.5	34
11	36
12	38
12.5	40
13	42
14	45
15	48
16	50
17	53
18	56
19	60
20	64
21	68
22	72
24	75
25	80
26	85
28	90
30	95
	100

* The series of rounded values would be valid also for multiples or submultiples of the given numerical values, e.g., 22 would be used, also 0.22; 2.2; 220; 2200, etc.

One of the greatest difficulties in the way of introducing the system of weight numbering is that a textile mill man associates a clear concept with specific numbers, e.g., with the English cotton yarn number 20 or the metric number 34, but not with the number "tex 30." An aid in conveying the new concepts would be a handy conversion table for the different types of yarn, a table which would show which tex numbers are equivalent to the yarn numbers used up to now. These tables should comprise: Yarn numbers in the metric, English, or any other system used up to now and their converted values; the calculated weight of these yarns, e.g., in g/km; the rounded numbers corresponding to this weight in a unit of the new international system of weight numbering, e.g., tex; and the deviation of the rounded tex numbers in per cent from the accurately calculated numbers, i.e., the weight in g/km. To illustrate this more clearly, Table II shows a section from an outline of a conversion table for cotton yarn numbers.

The conversion tables should comprise as many numbers as possible from the systems used heretofore, also from less customary ones, in order to meet all conceivable requirements. The deviations given in the last column, which result from the use of the rounded tex numbers, are very slight and practically negligible. This too shows that the use of numerical values other than those suggested in Table I is unnecessary for the purposes of yarn numbering, with the exception of a few special cases, which, however, will never be covered by standardization. Separate conversion tables should be set up for each type of yarn, and their use in practice would have the additional consequence that internationally the conversion of the old numbers into the new ones

TABLE II. Section from an Outline of a Conversion Table for Cotton Yarn Numbers

1 Metric No. (Nm)	2 English No. Ne_B	3 French No. (N_f)	4 Weight (g/1 km)	5 Proposed Tex	6 Deviation Tex: g/km (%)
33.87	20	16.93	29.53	30	+1.6
34	20.08	17	29.41	30	+2.0
35.56	21	17.78	28.12	28	−0.4
36.00	21.26	18	27.78	28	+0.8
37.25	22	18.63	26.84	26	−3.1
38.00	22.44	19	26.32	26	−1.2
38.95	23	19.47	25.68	26	+1.2
40	23.62	20	25.00	25	±0
40.64	24	20.32	24.61	25	+1.6

would be carried out uniformly; i.e., Ne_B 22, weight 26.84 g/km, is converted to tex 26 and not tex 27, which can be done without hesitation in consideration of the error, −3.1%. Consequently, the number 27 would no longer be used for yarns in the future.

If the two proposals discussed above should be accepted, i.e., (1) a rounded series with an interval between steps of 6%, corresponding to Table I, as a future general yarn number series; and (2) conversion tables for the individual types of yarn, which include as corresponding values for the old numbers only the numerical values of Table I in the weight numbering system, then we could strive for another international decision which would lead to a gradual introduction of the standard weight numbering. It should be recommended that spinners, while continuing to use their old numbers, should give the equivalent value of the new weight numbering system in parentheses, numbers which can be taken from the internationally adopted conversion tables. A cotton yarn with the English number 20 would be designated "20 (tex 30)"; a linen yarn with the English number 55 as "55 (tex 30)"; a cotton or linen yarn with the metric number 34 would be designated with the number "34 (tex 30)." This additional information would greatly facilitate the use of the new yarn numbers by the yarn consumer, since, by one glance at the information in parentheses on the labels, packages, bills, etc., he can learn the corresponding yarn numbers of different numbering systems and obtain simultaneously with the tex number the weight of the yarns in g/1000 m (the number in other corresponding units, for example, grex units—weight in g/10,000 m).

The above proposals have been sent by Germany to the Netherlands Standardization Bureau, the Secretariat for the Subcommittee on "Preferred Series of Yarn Numbers," with the request that they be considered by the subcommittee.

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Manuscript received November 9, 1954.

Felt from Man-Made Fibers[1]

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Introduction

Felt is defined [2] as a fabric built up by the interlocking of fibers by a suitable combination of mechanical work, chemical action, moisture, and heat. The word "felt" is most usually applied to structures prepared without spinning, knitting, or weaving and will be used in that sense in this paper. Nonwoven or pressed felt is one of the oldest forms of fabric. The Mongolian tribes of Asia were among its early users, and mention of it occurs in the writings of ancient Greeks and Romans. Legend ascribes the rediscovery of felt by Western man to a French monk of the eleventh century. The introduction of a mechanical process for making felt was accomplished in 1820 by J. R. Williams. This was indeed an important advance, for it eventually made possible the establishment of the modern felt industry and the production of precision felts for a variety of purposes. These include gaskets, seals, wicks, polishing wheels, filters, and antivibration mounts, as well as hats, apparel, and bootliners.

Only two major fibers have been used to make felts, namely, sheep's wool and the fur of other

[1] Based on a paper presented at the Gordon Research Conference on Textiles, New London, N. H., July 15, 1954.

animals. Nylon, rayon, and protein fibers have each been added to wool or fur felts in recent years, but have served mainly as additives in small proportions, imparting to the products some of their functional advantages or perhaps a cost advantage. Boeddinghaus [3] describes felts made from a mixture including a small proportion of stretched thermoplastic fibers. The major characteristics of all these felts were, however, determined by their major components, wool or fur.

Attempts have been made in the past to prepare felt or feltlike structures without wool or fur by adhesively bonding the fibers. Adhesion may be accomplished by the addition to the fibers of a binder. Bonding can also be achieved by the addition of fibers with a low softening temperature. These fibers become adhesive when activated by heat and/or pressure. Such materials have been described by Hoffman [8] by Boeddinghaus [3] and by Maxwell [9].

A felt must meet certain requirements of density, strength, resilience, and porosity, to name only a few, depending upon the end use for which it is intended. In some cases there is no need for a very strong or extremely dense felt; in others there is. It is, therefore, desirable to be able to prepare felts ranging from soft to hard and from relatively weak to very strong.

Wool Felt

The process for making wool felt, for example, is admirably suited to that purpose, for, by blending the proper wools in the right amounts and by controlling the intensity of the felting treatment, the density, strength, and hardness of the material can be varied. On examining a typical high-quality wool felt, one is impressed by certain features: it is a uniform assembly of fibers, made quite accurately to certain specifications of weight and thickness. It has certain minimum strength requirements. It may be quite hard, yet it will retain a certain amount of smooth flexibility in all directions when bent or twisted. The reason for this is the absence of bonding adhesion between fibers, such as would be imparted by addition of adhesives or bonding agents.

The only force that holds the entangled fibers together is friction. When external forces overcome some of this friction the fibers are relatively free to move past each other, a situation that would not exist in adhesively bonded materials. Thin felts would be expected to drape and be useful as apparel.

A further requirement of a high-quality wool felt is that it has adequate splitting resistance, that is, resistance to splitting in thickness, a property in which adhesively bonded fiber laminates are often deficient. As far as is known no material meeting all these requirements and encompassing the entire range of felt properties has ever been prepared heretofore without wool or fur as the major component.

In approaching the problem of making felt with fibers other than wool or fur as the chief constituent, one is immediately faced with the question: What is there about wool or fur fibers which makes them felt?

The wool felting process consists of three essential steps [1, 5]: (1) carding; (2) hardening; (3) fulling.

In the carding step the fibers are opened and formed into a sliver similar to the carding operation for yarns, except that the sliver is collected in open width on an accumulator apron until a batt has been built up. In the hardening step, the batts are passed between oscillating and vibrating plates in the presence of steam. This causes entanglement and interlocking of fibers, and the thickness is reduced considerably. The hardening operation is followed by fulling, usually carried out in fulling mills. In this type of mill, the hardened batt is pounded by hammers in the presence of lubricating agents, the effect being a shrinkage of the batt area and some increase in thickness. Certain finishing operations may follow the fulling. The initial weight of the batt and the extent to which the hardening and fulling steps are carried determine the density, hardness, and tensile properties of the felt.

During the hardening and fulling operations the wool fibers are subjected to heat and moisture, often in the form of steam; to pressure, rubbing, and impact; and to lubricants and other chemicals [4, 13]. When fibers other than wool or fur are subjected to similar treatments, the same type of felting is not obtained.

Numerous theories have been proposed to explain the felting of wool [10, 12, 14]. It appears to be the consensus that the following fiber properties are important (the order in which they are listed does not necessarily correspond to their importance): Crimp; curl; scales; ease of extension; recovery from extension; differential frictional coefficient.

Other factors which have been mentioned are [6, 7, 11, 12, 14]: Swelling; lubrication (absence of friction); plasticity (softening under heat, pressure, and moisture).

Felt of Man-Made Fibers

A. Man-Made Fibers in Wool Felting Process

The classical approach to a felt of man-made fibers would be to determine the properties of wool that are responsible for felting and then to select fibers with these properties or to try to build the properties into a special fiber. Attempts to do this in the past have not yielded the desired result, namely, felt made wholly from man-made fibers.

A modified approach was made in this laboratory to determine those properties responsible for the purely mechanical part of felting, the interlocking and entangling. For this modified approach, wool fibers were placed between two standard microscope slide glasses and observed under a low-power binocular microscope. By hand manipulation, the top slide glass was oscillated in a horizontal plane with a slight rotary motion. Downward pressure on the slide glass was sufficient to keep the glass in contact with the fibers. The motion used was similar to that of a laboratory-type Abbott felting machine. The test was repeated in the presence of water.

Through the microscope wool fibers were observed to move readily with or against each other. The whole mass of fibers seemed to be "alive." Individually, a particular fiber under observation seemed to move in one direction like a worm or snake. The fibers deformed readily. When water was introduced the "plasticity" of the fibers became quite marked and single wool fibers began to spiral, eventually ending up as a tight spiral. This occurred regardless of the direction of motion of the slide glass. These observations led to the conclusion that felting was a consequence of the elastic properties of wool.

This method offered a ready test for the screening of man-made fibers. Single fibers were placed under the microscope in a similar manner, and their behavior under the rubbing motion was studied. Dacron (Du Pont's polyester fiber) and Orlon (Du Pont's acrylic fiber) did not show movement similar to wool. A rubber filament of square cross section, however, could be seen to twist and tighten into a little ball, but returned to its original straight form as soon as the pressure on the slide was released.

TABLE I. Range of Physical Properties for Felting

	During Felting (Hot, Wet) Analogous to Rubber	After Felting (Room Temp., Dry) Analogous to Wool
Modulus (g/den)	<2	>10
Elongation (%)	>100	<50
Recovery from 20–30% strain (%)	>90	<50

The experiment was repeated using 2-in. lengths of rubber band. In a short while, with much twisting and turning, the rubber bands became a tight ball. Release of the slide pressure, combined with the elasticity of rubber, caused disentangling, but some extremely well-twisted and entwined sections remained entangled, even after removal of pressure.

These observations of rubber and wool and a knowledge of their respective physical properties suggested the following criteria, shown in Table I, for fiber properties. Wool may be thought of as having rubberlike elasticity under felting conditions. This rubberlike elasticity is probably responsible for the entanglement and entwining of the fibers. The scales may contribute more importantly to the permanence of the entanglement and to the shrinkage

The properties shown in Table I were used in scanning an array of available fibers. It was decided, finally, to pick one which met the conditions of rubberlike elasticity as defined in the table, namely, polyethylene fiber. During the felting test under the microscope polyethylene fibers formed spirals similar to those occurring with wool. Samples of polyethylene fiber were then run in the Abbott laboratory felting machine and formed felted structures. A polyethylene felt is shown in Figure 1. While this result indicated that felts could be formed during fulling.

from fibers other than wool, it also showed these remaining problems: (1) necessary refinement of fiber properties to yield a range of felts; (2) elucidation of conditions necessary to produce the required "elasticity" in other fibers.

From the vantage point of hindsight it is of interest to note here that the fundamental observations of the behavior of wool indicated that further work could center on felting process studies. In other words, instead of building the felting properties of wool into a man-made fiber and using this fiber in the conventional wool felting process, a revision of

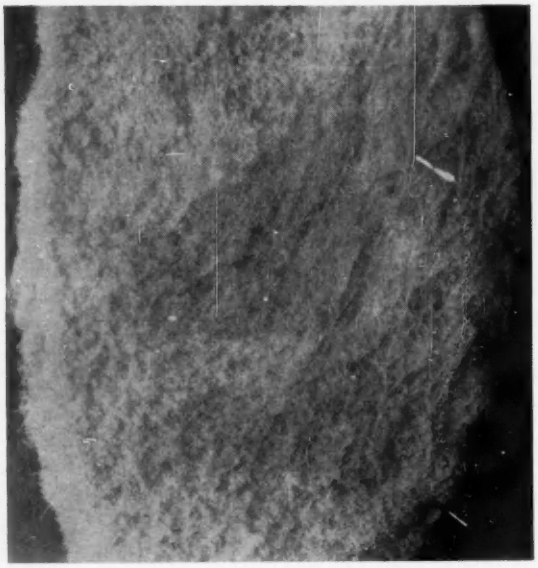

Fig. 1. Felt from polyethylene fiber.

the felting process itself seemed to be a more practical alternative.

B. New Process to Felt Man-Made Fibers

Additional work on the mechanical aspects of interlocking and entangling of fibers showed that carded batts could be sewed with thread and thereby acquired considerable laminar strength. This same effect was then obtained by passing carded batts through a needle loom which is commonly used in the carpet industry to secure loose fibers to a woven base by punching with multiple needles. Carpet underpads, made of inexpensive fibers, are produced in this manner. The needle loom has also been used to punch nylon fibers into a woven base for papermakers felts. Glass fibers and sisal fibers without a base fabric have been needle punched for use as reinforcement materials in laminates.

Thus, it was found that interlocking and entanglement could be produced in fibers that do not respond to the wool felting treatment. The needle loom contains a crosshead holding many barbed needles. The needles reciprocate up and down in such a way as to push some of the fibers through the batt. When the needles are withdrawn these fibers remain laterally extended through the batt thickness and promote entanglement of the otherwise parallel

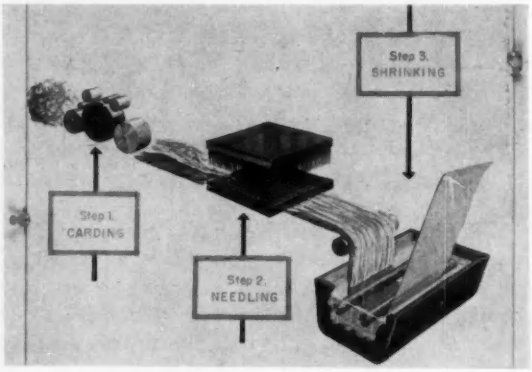

Fig. 2. Process for making felt from man-made fibers.

fibers. As the needle loom has been most commonly used, the batt is fastened to a base fabric during needle punching. No base fabric was used in this work.

In the shrinkage of wool felts during fulling, fibers migrate past each other in such a way as to decrease the area of the cloth. The fibers themselves may turn, twist, bend, and even stretch, but they probably do not sensibly change in denier. The shrinkage is, therefore, in the fabric, not the fiber. Man-made fibers can be produced with inherent shrinkage. When prepared by various special methods and then exposed to moisture, chemicals or heat such fibers shrink by increasing in denier and by decreasing in actual end-to-end length. It was reasoned that such fiber shrinkage, when induced in an entangled mass of fibers, would result also in shrinkage in area of the whole mass and thus compact the structure. Although Dacron polyester fiber has normally practically no inherent shrinkage, it can be specially manufactured so that it shrinks at temperatures near 100°C up to 50% or 75% of its length. It can be made in a variety of deniers and of accurately controlled shrinkages. Therefore, carded batts of shrinkable Dacron polyester staple were prepared, needle punched, and then exposed to temperatures around 100°C for shrinking. By the combination of steps outlined schematically in Figure 2, it was found that felt could be prepared from man-made fibers that do not have the physical properties prerequisite to the customary wool felting process as described in Table I. Moreover, felt produced from these man-made fibers by the combination of steps shown in Figure 2 not only has the minimum physical properties prescribed by the Society of Automotive Engineers

TABLE II. Strength Properties of Felt from Dacron *

SAE No.	Tensile Strength (psi)		Splitting Resistance (lb/2-in. width)	
	SAE Minimum	Dacron Actual	SAE Minimum	Dacron Actual
F-1	500	1712	33	45
F-5	400	1195	18	34
F-10	225	640	8	30
F-50	500	892	—	—

* Du Pont's trade mark for its polyester fiber.

TABLE III. Construction of Felt from Dacron *

SAE No.	Thickness (in.)			Weight (lb/sq yd)	
	Nominal	SAE Permissible Range	Dacron Actual	SAE Permissible Range	Dacron Actual
F-1	3/8	0.358–0.392	0.371	5.7 –6.3	6.4
F-5	3/8	0.350–0.399	0.355	4.36–4.82	5.0
F-10	3/8	0.341–0.408	0.348	3.03–3.35	3.0
F-50	0.075	—	0.074	1.12	0.98

* Du Pont's trade mark for its polyester fiber.

(SAE) but also surpasses them in all cases, as indicated in Table II, and possesses a number of additional advantages.

Properties of Felt from Man-Made Fibers

Once the felt has been made, it is stable to further shrinkage or felting. It can, therefore, be used where rubbing or impact occurs in the presence of moisture or lubricants. Felt from Dacron polyester fiber does not change density when so used because no further felting can take place. The fiber imparts its own properties to the material as a whole. Felt from Dacron polyester fiber is stronger, tougher, more durable, and has higher abrasion resistance under many conditions than wool felt. It is also resistant to mildew and fungi and to solvents, acids, or alkali to which the fiber is resistant.

After the specially prepared Dacron polyester fiber has been exposed to the shrinking conditions and optionally heat stabilized at 200°C for a short time, it will undergo no further shrinkage when again exposed to shrinking conditions. Therefore, felt made from shrinkable Dacron is quite stable at temperatures below that used for heat setting. The fiber shrinkage, the amount of needle punching, and the weight of the carded batt determine the characteristics of the felt—whether it will be hard or soft, and how dense and how strong it will be. Hence a range of fiber shrinkages is available to cover the entire range of felt properties.

Table III shows that felt of Dacron polyester fiber can be constructed to meet sizes and weights specified by SAE [15]. Because of the engineering flexibility inherent in man-made fibers, it seems reasonably certain that these felts can be made to numerous different specifications. Table III, for example, shows that as a first approximation the felts prepared are well within the thickness range specified by SAE [15] but fall slightly outside the listed weight range. It is obvious that in the second approximation slight adjustments can be made which will still leave the felts in the proper thickness range but will also bring them into the weight tolerance range. Figure 3 shows a typical stress-strain diagram obtained on an Instron tensile tester for a relatively thin but strong felt from Dacron polyester fiber. The felt weight was 13 oz/sq yd, and its thickness was 0.086 in. The test was performed in accordance

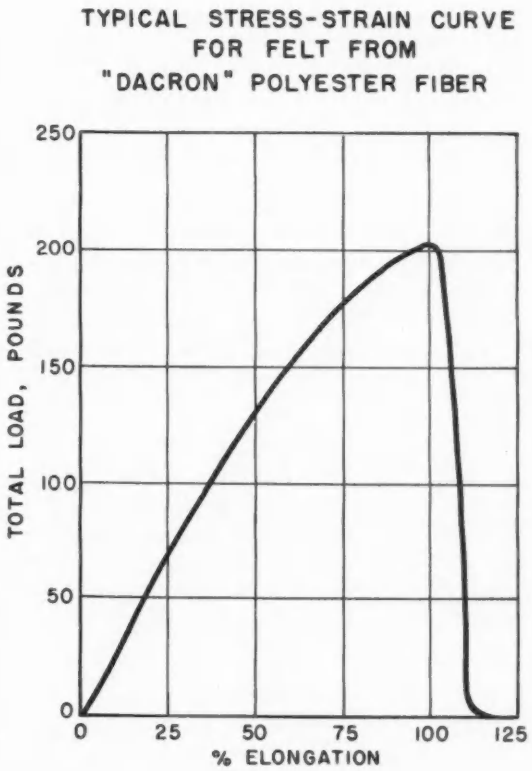

Fig. 3.

with the conditions prescribed in the ASTM tensile-test procedure for felts [2]. The total load at break for a 2-in.-wide specimen of 0.086 in. thickness corresponds to a tensile strength of 1220 psi. The elongation at break is about 100%. Figure 4 shows a representative record of a splitting resistance test. The felt used was a very dense specimen prepared from Dacron polyester fiber for use as a polishing felt. The weight was 8.7 lb/sq yd, and the thickness was 0.56 in. As prescribed by ASTM [2], the splitting resistance is measured by splitting the felt for a short distance in thickness along the middle third, inserting each of the lips so obtained in opposing jaws of a tensile tester, and completely pulling apart the felt. The average splitting resistance per 2-in. width is found from Figure 4 to be 44 lb.

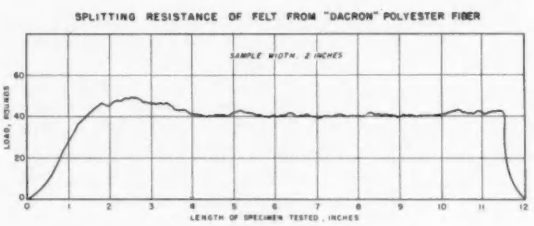

Fig. 4.

Uses for Felt from Man-Made Fibers

Felt from man-made fibers is expected to supplement wool felt and even replace it in applications where felt and fiber properties offer unique advantages. Some of these advantages have been mentioned: stability to further felting; resistance to heat, chemicals, mildew and fungi; exceptional strength; abrasion resistance. Several applications are listed in Table IV.

TABLE IV. Uses for Felts from Man-Made Fibers

Use	Advantage
Filtration	Chemical resistance; heat resistance; ease of cleaning
Wicking	Constant wicking rate; no density change
Polishing	Abrasion resistance; freedom from grit
Medical	Can be boiled; ease of cleaning, non-swelling; no allergy problems

Summary

Table V summarizes the finding that felts from man-made fibers can be prepared in a series of steps utilizing shrinkable fibers which produce effects similar to those of the wool felting process although the mechanism and principles by which they operate are completely different for steps 2 and 3.

TABLE V

Wool	Effect	Man-Made Fibers
1. Carding	⟶Uniformity⟵	1. Carding
2. Hardening	⟶Entanglement⟵	2. Needle punching
3. Fulling	⟶Stabilizing⟵	3. Shrinking

The method outlined above was found to provide the flexibility necessary to prepare felts from man-made fibers over a wide range of densities.

The alternative approach of using man-made fibers incorporating special properties which would make them operable in the wool felting process was explored. The approximate properties needed were determined, and felts from polyethylene fibers were prepared. It was found that this procedure was not nearly as versatile and as promising as the preferred method shown in Table V.

The process involving carding, needle punching, and shrinking is simple and lends itself to continuous operation. It should be easy to control because many if not all of the steps can be run dry and can be readily watched. The fiber is clean and ready to use as received. Consequently, the felt product is reproducible to a high degree of accuracy. In addition, a choice of fiber denier is possible. Tests have already indicated that heavier-denier fibers make superior plate-glass polishing felts.

Although most of the present work has been performed with Dacron polyester fiber, other man-made fibers are being investigated in this process. These results will be reported at a later date.

Felt of man-made fibers is expected to supplement wool felt in some applications and to replace it in many. Even more important, it will become a new material of construction extending the use of felt into new fields and applications by virtue of higher strength, greater heat stability, greater chemical resistance, and resistance to mildew, moths, and fungi.

Acknowledgments

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International Congress

of

Scientific Research Applied to the Textile Industry

In connection with the Second International Textile Exhibition in Brussels, which is to be held from June 25 to July 10, 1955, one of the most important events is to be an International Congress of Scientific Research in the Textile Industry.

The Congress will take place on June 27, 28, and 29, 1955. Three important lectures of general interest will be given. There will, in addition, be papers presented by scientists of Belgium and from other countries relating progress to date in their specific fields of textile research.

All inquiries concerning this Congress are to be addressed to the Secretary of the Congress, c/o Centre Scientifique et Technique d l'Industrie Textile Belge, 24, rue Montoyer, Brussels, Belgium.

Cotton Quality Study

III: Resistance To Soiling

Nelson F. Getchell

Technical Department, National Cotton Council of America

I. The Problem and Its Opportunities
II. The Technical Aspects of the Problem
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The Solid Component
Particle Size
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The Forces of Impingement
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2. Test Methods
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I. The Problem and Its Opportunities

The importance of research directed toward improved methods of maintaining the cleanliness and appearance of textile materials during use can hardly be overestimated. In the United States commercial laundries alone handle over 2 million tons of fabric a year, more than 90% of which is cotton—the equivalent of 15 billion yards of 80-square print cloth.

Of equal concern is the tremendous effort expended on such household fabrics as rugs, upholstery, and draperies where laundering is usually difficult and sometimes impossible. In these, too, cotton holds a significant share of the market. For instance, in just the last few years, the use of cotton carpets and rugs has grown until in 1953 it represented roughly one-third of the total yardage of the soft floor coverings produced. Almost one-half of the upholstery field and about 70% of the drapery market are held by cotton.

[1] Previous papers in this series appeared in April and November 1949.

Most of the effort to cope with fabric soiling has, up to now, been directed toward the development of better cleaning agents, methods, and machines. However, there remains a relatively untouched area for research which offers a new approach to the problem and a challenge to the combined efforts of the textile and chemical industries, that is, the building of a "resistance to soiling" into the fabric. It involves not only the quality of "resistance" but also that of a "greater ease of removal."

The purpose of this report is to review the present knowledge in the field, indicating the potential markets for improvement; to discuss the technical factors involved; and to point out some unanswered questions with suggestions for further research.

Effects of Soil

Dirt is a menace to health; it detracts from the comfort of garments and the appearance of all fabrics. It shortens the useful life of materials through abrasive wear and the action of frequent cleaning operations. Of these, changes in appearance always

has been and still is the most important factor to the consumer. Comfort and sanitation are important mainly in those items that come in close contact with the body, while the decrease in fabric life expectancy is often overlooked for lack of a means of comparison.

Importance of Appearance

The serviceability of many items is more often judged by appearance than by actual failure. The loss of luster and change in color which may result from soiling and cleaning frequently determine the the useful life of an article. Even where health and comfort are important, degree of sanitation and cleanliness is usually measured by appearance. "Eye appeal" almost always forms the yardstick by which one judges the degree of soiling, the adequacy of removal, the hygienic value, and the extent of wear in a textile fabric. Thus, appearance is inextricably associated with any discussion of soiling and soil resistance.

Cotton fabrics, because of their multitude of end uses, are subject to a wider range of soiling conditions than any other textile material. Since cottons are so often employed in the undyed or bleached state, where dirt and stains are readily visible, they have become closely associated in the consumer mind with the need for frequent cleaning. They have thus acquired the reputation for being more easily soiled than other materials. Also supporting this belief is the widespread use of cotton for sanitary purposes and as cleaning materials such as handkerchiefs, wiping cloths, and diapers, where conditions of contamination are extreme.

Cotton is the logical material for such uses because of its excellent launderability as well as its low cost. Its high wet strength, durability, and resistance to alkalinity instituted the custom of cleaning cotton fabrics under more severe conditions than could be used for wool and rayon goods. The consumer has thus learned to expect from all cotton materials that fresh, clean appearance associated only with laundering.

Types of Soil

Many factors which affect the appearance, comfort, and usefulness of a fabric can be included under the broad heading of soiling. Thus, wrinkling or loss of freshness and crispness detract from appearance; while invisible soils such as odors and colorless salts of perspiration lower the comfort and hygienic value. Such highly important qualities can be restored only by laundering or dry cleaning.

However, the form of soiling that presents the greatest problem and the one to which all fabrics are subject is the gradual change in appearance—the progressive darkening, discoloration, and loss of luster—caused by the complex mixture commonly known as dirt. In varying degree and form, it is found everywhere—in the home, at work, and at play. When uniformly distributed over the surface, it is called dirt or soil; when confined to a small area it is termed a spot, which, if extremely difficult or impossible to remove, becomes a stain. In the air it is called dust; when mixed with water, it is mud; and when mixed with oil or grease from food, machinery, or the skin, it is referred to as grime.

This form of soil, especially when finely divided and deeply imbedded presents the most serious obstacle to fabric maintenance. The many devices—brushes, brooms, vacuum cleaners, etc.—designed to remove it are evidence of the effort expended to combat the problem. In some cases even laundering cannot completely remove the last trace of ingrained soil that builds up over a period of time.

In the final analysis, the serious problem is not with the loosely held particles that can be removed easily by simple mechanical means—shaking, brushing, wiping, or sweeping—but with the dirt that still remains in the material after these operations have been carried out. Also in the case of items which require laundering because of other factors of freshness or sanitation, the quickly soluble or easily emulsifiable substances present no problem. It is the difficultly removed portion and the redeposited soil, building up over a period of cleaning or washing, that cause the progressive deterioration in appearance and necessitate the severe methods of removal.

Soil Resistance

It is difficult to define in simple terms the meaning of "soil resistance." The ultimate is, of course, the complete prevention of all forms of fabric contamination, an obviously unattainable goal at present. A means for preventing or slowing down the change in appearance due to soil provides a more realistic approach, one that may be accomplished either by camouflaging the effects of dirt, as practiced for generations through the use of color and color pattern, or by the more direct method of reducing soil reten-

tion. In the latter sense, "resistance to the retention of soil" is a more descriptive term than soil resistance. It also becomes synonymous with "greater ease of soil removal" when it is considered that only the dirt which cannot be removed by simple methods of cleaning presents a problem.

Stated mathematically, the dirt retained by a fabric is equal to the amount impinging on the material minus the amount removed by periodic cleaning (Retention = Impingement − Removal). Retention can be lowered either by increasing the amount of dirt removed, or by reducing the quantity that comes into contact with the fabric. Since the latter involves control of the environment, an increase in removal remains the only practical possibility. The principle of soil resistance is aimed at increasing the efficiency of removal, not by an improvement in the cleaning method itself but through a change in fabric characteristics to allow dirt to be more easily removed by a simple cleaning operation.

A "simple" method of soil removal varies with the end use, the structure of the fabric, and the habits of the consumer. For instance, vacuum cleaning, which is commonly used on rugs and upholstery, cannot be considered a suitable or simple method for cleaning such items as dresses, shirts, or table linens. Here, only a gentle shaking or brushing is possible. On the other hand, in the extreme case of baby diapers where other factors far outweigh appearance, laundering is the only acceptable method.

Market Analysis

It is soon evident that an attack on the problem must be preceded by a study of consumer habits and preferences and a careful analysis of the end uses and methods of fabric maintenance. Such a market analysis has been carried out. It shows that, while resistance to soiling is a desired quality in a large number of uses, in only a few is the current demand strong enough to warrant even a moderate increase in cost Although few in number, those uses in which it is important represent sufficient volume to constitute a real incentive for active research. Too, success in these fields should generate more active demand in others and provide an expanding market for the future.

A breakdown of fabric uses into three broad groups, as shown in Tables *A*, *B*, and *C*, is helpful in determining where and to what extent demand now exists. The first and most important group includes those items where discoloration from dirt is common, where appearance or "eye appeal" rather than sanitation is of greatest concern, and where cleaning is difficult and laundering often impossible. Rugs and carpets, upholsteries, draperies, and automobile fabrics are typical of these largely household, institutional, and semi-industrial uses.

The greatest common denominator making this group an outstanding problem is the difficulty or impossibility of applying ordinary wet-cleaning methods. Elimination of the need for cleaning by these means is of primary interest to the consumer. Reduction in fabric soil retention and increase in the ease of dirt removal by simple, in-place, mechanical cleaning operations—shaking, brushing, sweeping, or vacuum cleaning—should be the goal of research.

In the second group of end uses appearance is again the major consideration, but with sanitation also highly important. It includes most outer clothing—women's and children's dresses, suits, and skirts; men's jackets, shirts, and trousers; sport clothing; aprons and nurses' uniforms—garments that are exposed to the public view rather than the seldom-seen underclothing. The factor of wrinkling and mussing, together with the desire for a "fresh" appearance, are often more important in determining the frequency of cleaning than actual discoloration from dirt. Wrinkle-resistant finishes have thus found their widest application in these items, and can in themselves be considered soil-resistant treatments. Although odor and perspiration are also a problem, they are not the most important consideration as in the case of the more personal undergarments.

For items in this second class, dirt is only one of many factors influencing appearance. Such qualities as drape, hand, and luster are often of far greater importance to the consumer than soil resistance. While resistance to visible soil is also a desirable fabric characteristic, treatments must not otherwise adversely effect the even more important values. This group presents a latent rather than an active demand for soil-resistant treatments.

The third large end-use category includes such items as sheets and pillowcases, towels and bathmats, underwear, socks, diapers, and handkerchiefs—articles that come into close daily contact with the body. Although often measured by appearance, the comfort and sanitary values of these personal items are of primary importance to the consumer. Invisible forms of soil—odors, perspiration, etc.—are common.

(Text continued on page 157.)

TABLE A. ***Group I:*** **Markets in Which Improvements in Resistance to Soiling Are of Most Importance** [a]

Item	Estimated Size of Market [b] (1000 lb)			Estimated Per Cent of Market Held by Cotton [b]
	Cotton [c]	Other Materials [d]	Total Cotton and Other Materials	
HOUSEHOLD				
Bedspreads				
Tufted				
Fabric	19,763	—	19,763	100
Yarn	20,902	—	20,902	100
Woven				
Jacquard	18,770	2,086	20,856	90
Other	7,443	392	7,835	95
Curtains				
Lace	2,955	521	3,476	85
Other				
Cottage	6,102	4,068	10,170	60
Ruffled	12,187	28,437	40,624	30
Tailored	1,656	14,908	16,564	10
Drapery, upholstery, and slip-cover fabric, not elsewhere classified	128,664 [e]	111,073 [e]	239,737 [e]	54
Laces, including tablecloths and scarfs	3,516	992	4,508	78
Rugs and carpets				
Cotton, excluding scatter rugs, bath mats and sets 4 by 6 ft or smaller				
Tufted	23,778 [f]	5,945 [f]	29,723 [f]	80
Woven and knitted	9,035	475	9,510	95
Wool, excluding automobile carpeting	[f]	156,000 [f]	156,000 [f]	
Slip covers, furniture	16,909	1,879	18,788	90
Stamped art for embroidery	2,545	315	2,860	89
Window shades	20,142	[g]	20,142 [g]	[g]
Total Household	294,367	327,091	621,458	47
INDUSTRIAL				
Automobile				
Covers, seat				
Seat and backing fabric	1,478	72,416	73,894	2
Seat backing and facing	9,488	6,325	15,813	60
Linings and upholstery fabric				
Passenger cars	36,480	93,804	130,284	28
Trucks	4,909	—	4,909	100
Tops, convertible, excluding replacement tops	1,031	1,163	2,194	47
Awnings	27,128	[g]	27,128 [g]	[g]
Flags	1,647	[g]	1,647 [g]	[g]
Luggage linings	1,679	6,714	8,393	20
Umbrellas				
Beach, garden, and tractor	566	—	566	100
Hand	39	925	964	4
Venetian blind tape	3,879	1,662	5,541	70
Total Industrial	88,324	183,009	271,333	33
GRAND TOTAL	382,691	510,100	892,791	44

[a] These items represent markets in which soiling is a major consideration in that these items are not laundered regularly—if at all. These items represent the strongest potential market for a soil-resistant finish.

[b] Based on data contained in *Cotton Counts Its Customers* (preliminary 1953 estimates), National Cotton Council of America.

[c] Estimated gross weight of fabric and/or yarn in woven and/or knit products.

[d] Estimated gross weight of fabric and/or yarn in woven and/or knit products in terms of cotton equivalent gross weight.

[e] Excludes backing material of pile fabric where a soil-resistant finish is unimportant.

[f] Excludes backing material of cotton and wool rugs where a soil-resistant finish is unimportant.

[g] Represents only cotton's share of the market. Sufficient data were not available to estimate total markets.

TABLE B. ***Group II:*** **Markets in Which Resistance to Soiling Are Desirable**

Item	Estimated Size of Market [a] (1000 lb)			Estimated Per Cent of Market Held by Cotton [a]
	Cotton [b]	Other Materials [c]	Total Cotton and Other Materials	
MEN'S AND BOYS' APPAREL				
Bathrobes, dressing gowns, and smoking jackets	2,191	4,253	6,444	34
Coats and jackets				
Blanket and sheep-lined				
Men's	10,857	20,163	31,020	35
Boys'	3,919	7,279	11,198	35
Mackinaws and meltons				
Men's	2,363	7,087	9,450	25
Boys'	992	2,974	3,966	25
Overcoats and topcoats				
Men's and students'	388	19,005	19,393	2
Boys'	52	2,532	2,584	2
Separate coats				
Men's	5,789	13,507	19,296	30
Boys'	694	2,778	3,472	20
Jackets, including leather				
Men's	13,636	11,156	24,792	55
Boys'	4,248	5,192	9,440	45
Gloves, dress and semidress	602	1,807	2,409	25
Overalls and coveralls				
One-piece suits	13,360	—	13,360	100
Overall jackets	7,208	—	7,208	100
Overalls				
Men's	31,312	—	31,312	100
Boys'	4,324	—	4,324	100
Rainwear, dress and utility, excluding plastic	4,659	1,812	6,471	72
Shirts				
Dress				
Men's	46,192	1,925	48,117	96
Boys'	3,723	76	3,799	98
Polo, "T," and basque				
Men's	43,257	437	43,694	99
Boys'	11,282	114	11,396	99
Sport, woven				
Men's	39,345	32,192	71,537	55
Boys'	12,927	1,436	14,363	90
Sweat				
Men's	8,400	260	8,660	97
Boys'	2,875	—	2,875	100
Work				
Men's	34,135	697	34,832	98
Boys'	2,103	21	2,124	99
Suits				
Dress				
Summer weight				
Men's	1,065	10,765	11,830	9
Boys'	370	370	740	50
Winter weight				
Men's	2,416	45,899	48,315	5
Boys'	268	5,095	5,363	5
Riding, hunting, and camping	9,603	1,695	11,298	85
Slack				
Men's	1,354	903	2,257	60
Boys'	652	163	815	80

[a] Based on data contained in *Cotton Counts Its Customers* (preliminary 1953 estimates), National Cotton Council of America.
[b] Estimated gross weight of fabric and/or yarn in woven and/or knit products.
[c] Estimated gross weight of fabric and/or yarn in woven and/or knit products in terms of cotton equivalent gross weight.
[d] Represents only cotton's share of the market. Sufficient data were not available to estimate total market.

TABLE B (*Continued*)

Item	Estimated Size of Market [a] (1000 lb) Cotton [b]	Other Materials [c]	Total Cotton and Other Materials	Estimated Per Cent of Market Held by Cotton [a]
MEN'S AND BOYS' APPAREL (*Cont.*)				
Sweaters				
Men's	1,004	9,039	10,043	10
Boys'	391	1,172	1,563	25
Trousers				
Dress and sport				
Men's	6,309	46,263	52,572	12
Boys'	10,063	6,484	16,547	61
Work				
Dungarees				
Men's	70,391	711	71,102	99
Boys'	36,694	371	37,065	99
Other				
Men's	77,197	1,575	78,772	98
Boys'	2,656	54	2,710	98
Washable service apparel				
Bakers', cooks', and barbers'	3,457	71	3,528	98
Medical and hospital	3,661	153	3,814	96
Work aprons, industrial	5,512	[d]	5,512 [d]	[d]
Subtotal, Men's and Boys' Apparel	543,896	267,486	811,382	67
WOMEN'S, MISSES', AND JUNIORS' APPAREL				
Aprons, smocks, pinafores, and hoovers	8,959	3,839	12,798	70
Bathrobes and beach robes	9,342	4,601	13,943	67
Blouses, waists, and shirts	21,603	20,755	42,358	51
Coats and jackets				
Coats				
Fur trimmed	80	3,920	4,000	2
Without fur trim	3,336	63,378	66,714	5
Jackets	1,370	2,333	3,703	37
Dresses				
House	65,068	10,592	75,660	86
Street	28,885	74,274	103,159	28
Other knit, including suits and skirts	92	1,744	1,836	5
Gloves and mittens, dress and semidress	1,356	2,880	4,236	32
Housecoats, including breakfast and brunch coats	6,629	1,870	8,499	78
Negligees and bed jackets				
Bed jackets	56	564	620	9
Negligees	445	1,579	2,024	22
Overalls and coveralls	1,531	31	1,562	98
Rainwear, excluding plastic	1,199	6,793	7,992	15
Sportswear				
Halters	231	23	254	91
Playsuits, sunsuits, etc.	9,039	377	9,416	96
Polo, "T," basque, and other knit shirts	9,925	203	10,128	98
Slacks	1,960	3,338	5,298	37
Slack suits	42	280	322	13
Suits and skirts				
Skirts	16,908	35,928	52,836	32
Suits	857	20,577	21,434	4
Sweaters and jerseys				
Coat and cardigan sweaters	636	15,276	15,912	4
Pullovers and jerseys	492	15,909	16,401	3
Washable service apparel				
Nurses' uniforms	3,941	1,609	5,550	71
Other washable service apparel	3,482	520	4,002	87
Subtotal, Women's, Misses' and Juniors' Apparel	197,464	293,193	490,657	40

TABLE B (*Continued*)

Item	Estimated Size of Market [a] (1000 lb) Cotton [b]	Other Materials [c]	Total Cotton and Other Materials	Estimated Per Cent of Market Held by Cotton [a]
CHILDREN'S AND INFANTS' APPAREL				
Bathrobes and beach robes				
Girls' and teen-age girls'	1,218	78	1,296	94
Children's and infants'	1,417	354	1,771	80
Blouses, waists, and shirts				
Girls' and teen-age girls'	1,850	584	2,434	76
Children's and infants'	1,583	347	1,930	82
Coats, jackets, legging sets, ski and snow suits and pants				
Coats				
Girls' and teen-age girls'	443	8,421	8,864	5
Children's and infants'	523	4,710	5,233	10
Coats, leggings, and legging sets				
Girls' and teen-age girls'	209	836	1,045	20
Children's and infants'	1,580	4,741	6,321	25
Jackets, except leather				
Girls' and teen-age girls'	414	431	845	49
Children's	1,262	443	1,705	74
Ski and snow suits				
Girls' and teen-age girls'	137	279	416	33
Children's	309	926	1,235	25
Toddlers' and infants'	421	818	1,239	34
Ski and snow pants				
Girls' and teen-age girls'	10	332	342	3
Children's and infants'	33	141	174	19
Dresses				
Girls' and teen-age girls'	13,841	2,442	16,283	85
Children's	13,037	832	13,869	94
Toddlers' and infants'	5,622	1,071	6,693	84
Gloves and mittens	82	459	541	15
Overalls and coveralls				
Knit	3,361	104	3,465	97
Woven				
Girls' and teen-age girls'	7,208	—	7,208	100
Children's	18,959	192	19,151	99
Toddlers'	1,197	—	1,197	100
Infants'	2,400	24	2,424	99
Rainwear				
Girls' and teen-age girls'	67	190	257	26
Children's	79	141	220	36
Sportswear				
Playsuits, sunsuits, and shorts				
Girls' and teen-age girls'	6,308	64	6,372	99
Children's	9,173	187	9,360	98
Toddlers'	1,643	34	1,677	98
Polo, "T," basque, sweat and other knit shirts				
Children's	11,199	—	11,199	100
Infants'	2,303	—	2,303	100
Slacks				
Girls' and teen-age girls'	821	232	1,053	78
Children's	3,780	615	4,395	86
Slack suits				
Girls' and teen-age girls'	13	52	65	20
Children's	679	226	905	75
Suits and skirts				
Skirts				
Girls' and teen-age girls'	1,124	1,268	2,392	47
Children's and toddlers'	439	494	933	47

TABLE B *(Continued)*

Item	Estimated Size of Market [a] (1000 lb) Cotton [b]	Other Materials [c]	Total Cotton and Other Materials	Estimated Per Cent of Market Held by Cotton [a]
CHILDREN'S AND INFANTS' APPAREL *(Cont.)*				
Suits and skirts *(Cont.)*				
Suits, except ski and snow				
Girls' and teen-age girls'	153	803	956	16
Girls' (6X and under)	96	360	456	21
Boys' (6X and under)	332	271	603	55
Wash suits				
Boys'	1,421	271	1,692	84
Toddlers'	898	100	998	90
Sweaters, jerseys, and pullovers				
Girls' and teen-age girls'	239	2,146	2,385	10
Children's	873	3,491	4,364	20
Infants'	209	1,885	2,094	10
Subtotal, Children's and Infants' Apparel	118,965	41,395	160,360	74
Total Apparel	860,325	602,074	1,462,399	59
HOUSEHOLD				
Retail piece goods, apparel	96,180	75,570	171,750	56
Tablecloths and napkins, excluding linen				
Damask	6,443	—	6,443	100
Other than damask				
Cotton	12,955	—	12,955	100
Synthetic and mixtures	762	2,287	3,049	25
Thread for home use				
Cotton				
Crochet and hand knitting	6,303	—	6,303	100
Darning	128	—	128	100
Embroidery	601	—	601	100
Sewing	4,953	—	4,953	100
Other cotton	2,243	—	2,243	100
Synthetic	—	130	130	—
Total Household	130,568	77,987	208,555	63
INDUSTRIAL	—	—	—	—
GRAND TOTAL	990,893	680,061	1,670,954	59

Comfort in contact with the body, freedom from odor, absorbency, and freshness are necessary qualities that can be maintained only through frequent contact with soap and water. Weekly, and in many cases daily, laundering is a well-established custom. Even if a treatment could be found to prevent and reduce visible soiling, there is doubt that this consumer habit could or should be changed.

Here, the greatest need has always been for methods to improve soil removal and reduce redeposition during laundering. These problems, however, are now receiving a large share of research attention which has already resulted in greatly improved detergents and laundering equipment. To be of economic value, further improvements in this direction require a low initial cost and considerable consumer education. It must be demonstrated that any increase in cost is warranted not only because of improved appearance but also because of longer fabric life through a reduction in the need for the severe laundering methods that now tend to progressively damage these materials.

TABLE C. ***Group III:*** **Markets in Which Improvements in Resistance to Soiling Are of Secondary Importance** [a]

Item	Estimated Size of Market [b] (1000 lb) Cotton [c]	Other Materials [d]	Total Cotton and Other Materials	Estimated Per Cent of Market Held by Cotton [b]
MEN'S AND BOYS' APPAREL				
Handkerchiefs, excluding imports	11,628	612	12,240	95
Hosiery	53,202	17,734	70,936	75
Pajamas	34,826	1,077	35,903	97
Underwear	103,117	5,427	108,544	95
Subtotal, Men's and Boys' Apparel	202,773	24,850	227,623	89
WOMEN'S, MISSES', AND JUNIORS' APPAREL				
Foundation garments	20,951	20,130	41,081	51
Handkerchiefs, excluding imports	2,327	582	2,909	80
Hosiery	12,176	74,793	86,969	14
Nightgowns, pajamas, and nightwear	26,376	24,348	50,724	52
Underwear	25,645	109,327	134,972	19
Subtotal, Women's, Misses', and Juniors' Apparel	87,475	229,180	316,655	28
CHILDREN'S AND INFANTS' APPAREL				
Diapers, excluding paper	22,907	—	22,907	100
Hosiery	13,006	542	13,548	96
Infants' wear, misc.	570	485	1,055	54
Overalls and coveralls	33,111	334	33,445	99
Pajamas and other nightwear	24,339	753	25,092	97
Underwear	33,468	13,670	47,138	71
Subtotal, Children's and Infants' Apparel	127,401	15,784	143,185	89
Total Apparel	417,649	269,814	687,463	61
HOUSEHOLD				
Blankets and blanketing	50,060	26,955	77,015	65
Comforters and quilts	2,097	7,435	9,532	22
Curtains, shower	167	8,207	8,374	2
Mattress covers	4,252	472	4,724	90
Pads, quilted	4,213	43	4,256	99
Pillowcases and tubing	35,371	—	35,371	100
Sheets	198,755	2,008	200,763	99
Towels and toweling	132,495	8,457	140,952	94
Total Household	427,410	53,577	480,987	89
INDUSTRIAL				
Medical supplies and equipment				
Adhesive tape and plaster	3,378	[e]	3,378	[e]
Bandages, gauzes, and sponges	27,011	—	27,011 [e]	100
Sanitary napkins	19,571	[e]	19,571 [e]	[e]
Total Industrial	49,960	—	49,960	100
GRAND TOTAL	895,019	323,391	1,218,410	73

[a] These items represent markets in which soiling is not a primary consideration in that these items are laundered regularly. Generally, these items do not represent a potential market for a soil-resistant finish.

[b] Based on data contained in *Cotton Counts Its Customers* (preliminary 1953 estimates), National Cotton Council of America.

[c] Estimated gross weight of fabric and/or yarn in woven and/or knit products.

[d] Estimated gross weight of fabric and/or yarn in woven and/or knit products in terms of cotton equivalent gross weight.

[e] Represents only cotton's share of the market. Sufficient data were not available to estimate total market.

II. The Technical Aspects of the Problem

A glance at the voluminous literature on detergency is proof enough of the study that has gone into this phase of the subject. Although important, it is only one of the several possible approaches to the problem. Except for the extensive theories developed to explain the removal of dirt by soap and water, there is little organized information concerning the other mechanisms involved in soiling. A better knowledge of the individual components making up the fiber-soil complex is fundamental to an understanding of its formation and stability. As one writer [86] has worded it, "Literature references to the nature of soils and the manner in which they are attached to fabrics are relatively lacking. As one reviews the past work on detergency it is apparent that, all too often, insufficient consideration has been given to the nature of the soil that is to be deterged from a surface and even to the nature of the surface itself. Yet a proper understanding of soils and soiling is essential to a thorough understanding of the overall detergent process."

1. The Nature of Soil

The accumulation of dirt resulting from the everyday use of clothing and household textiles is always a mixture, the components of which vary according to the factors of environment, fabric use, and customs of the individual. With such an infinite number of variables, to list all possible soil types and combinations would be an endless task. Luckily, for an understanding of the problem, this complex mixture can be considered under two general categories—a fluid component, usually an oil or grease; and a solid component made up of small, more or less inert particles. That the dirt accumulated by clothing and household fabrics is usually a mixture of these two is evident not only from the few analyses that have been made but also from everyday personal experience. Further, it is obvious that these components of natural dirt, although in themselves complex mixtures, originate in large part from the common substances encountered in daily living.

To study the nature of these mixtures, dirt from several different sources has been examined. Ordinary street sweepings, rug dirt picked up by vacuum cleaners, and the accumulations from air filters have been analyzed with the idea that they are typical of the various soils encountered by textile materials and that they would, therefore, be useful in constructing a synthetic dirt for artificial soiling purposes. An analysis of the oily constituents of naturally soiled clothing has also been made.

Although a knowledge of the chemical composition of natural dirt should prove to be a useful tool in helping to understand the nature of fabric soil, an important point to keep in mind is the fact that street sweepings or dirt picked up in vacuum cleaners may not necessarily have the same composition as the nonremoved soil remaining in a fabric after cleaning. With rugs, for instance, it is not the dirt that is removed that presents the problem; it is the material remaining after vacuum cleaning that causes the gradual change in appearance over a period of time. Because of the obvious technical difficulties involved, it is not surprising that only scattered information is available concerning the characteristics of this more tenaciously held soil.

What little has been done [85] through spectrographic and chemical analyses indicates that the same elements and ratios of elements are present in both mechanically removed and nonremoved dirt. However, there remains room for doubt since the experiments were carried out with artificial soiling methods and synthetic soil mixtures which may differ radically from natural dirt accumulated under normal service conditions. For instance, it has been shown [112] that a natural carpet dirt and a synthetic soil (similar to the one shown in Table V) could both be removed with equal ease from a carpet when artificially embedded; but that neither coincided with the soil-removal characteristics of a carpet exposed to actual service. This points to the still unanswered question concerning the difference between the easily removable and the difficultly removable dirt found in fabrics soiled by actual use. Do these fractions differ only in relative quantity and particle size distribution, or is there a further distinction between them in some factor of chemical or physical composition? Further research is needed to answer this question.

The Fluid Component

Aside from its own presence, the most important soiling function of the fluid constituent is its tendency to pick up and retain solid particles. For instance, everyone is familiar with the fact that a spot of butter or salad oil on a fabric, unless immediately removed, soon becomes dirtier and more discolored.

This ability to bind solid particles has been termed "oil bonding" to which a large part of fabric soiling can be ascribed.

The possible sources of oily fabric soil are many. Incompletely burned liquid fuels from industrial plants and home heating units, as well as the greasy vapors from cooking, continually settle out of the air directly onto fabrics or other exposed surfaces that may later come into contact with fabrics. It has been estimated that 40 lb of such oily liquids are volatilized in the average home over a period of a year. Lubricating oils and greases from automobiles and machinery also provide a common source of fabric contamination, as do the excretions of the human skin and the oils and fats from food.

The chemical nature of the oily materials, their degree of saturation and film-forming capacity is of considerable importance in respect to their ease of removal after a period of time. For instance, quick-drying vegetable oils, such as linseed or tung, form solid films in a matter of hours, whereas more fully saturated oils may take days or weeks to effect a change. Thus, unsaturated oils or polymerizable substances and the solids bound by them become progressively more difficult, or even impossible to remove because of this chemical change in the soil itself.

Although oils and greases are the most common of the nonsolids found in fabric soil, it is not necessarily restricted to them. Other viscous or sticky substances and nonvolatile liquids such as tars, gums, and resins would also be included in this category.

While considerable effort has gone into developing various mixtures thought to be representative of the oily component, only a few attempts have been made to determine either the relative quantity or chemical composition of the oily substances actually present in soiled fabrics. As would be expected, however, what little has been done shows a wide range of variation. An analysis of such domestically soiled personal items as cotton shirts, linen collars, pillowcases, hand towels, and wool socks gave a range of 0.25% for shirts to 1.2 on collars, based on the weight of the fabric [16]. Table I shows the average chemical composition of the oily matter extracted from these articles. It seems only logical that these should bear a close resemblance to the oils and fats excreted by the human skin.

Samples of vacuum cleaner dirt taken from rugs over a wide geographical area contained an average of 3% extractable material, with some ranging as high as 7½% [112]. An examination of the solvent extract showed, in addition to oil and grease, the presence of such substances as rubber, tar, asphalt, waxes, gums, and resins. Analysis of ordinary street dirt gave an ether-soluble component ranging from 5% to almost 13%; while that of air-borne soil

TABLE I. Average Composition of Oily Material from Domestically Soiled Articles *

Chemical Nature	Approximate Percentage
Free fatty acids (C_{18}; I.V. = 46)	32
Triglycerides of higher fatty acids (C_{18}; I.V. = 46)	29
Fatty alcohols and cholesterol (mol. wt. 275; I.V. = 63)	15
Short-chain fatty acids (butyric, caproic, caprylic, etc.)	3
Saturated and unsaturated hydrocarbons (C_{20}; I.V. = 50)	21

Note: I.V. = iodine value.

* Source: *Research* [16].

TABLE II. Analysis of Air-Borne Dirt *

Dirt Fraction	From Dry Filter (%)	From Water-Washed Filter (%)
Ether soluble (1) †	22.1	1.3
Hot-water soluble (2)	14.2	6.3
Hot dilute HCl soluble (3)	11.2	33.3
Loss on ignition of insoluble residue (4)	32.8	15.4
Acid-insoluble inorganic residue (5)	19.7	43.7
Total organic matter (1) and (4)	54.9	16.7

* Source: *Soap and Sanitary Chemicals* [49].

† Snell [109] has analyzed the possible sources of the above as follows:

1. The oils extracted with ethyl ether may consist of saponifiable oils, mineral oils, waxes; and nonsaponifiable fatty matter such as sterols and true waxes. In some locations a large part of this fraction could consist of incompletely burned hydrocarbons.
2. Materials dissolved out by cold water may consist of sugars, salts, soluble hydroxides, etc. That soluble only in hot water may consist of difficultly soluble salts, oxides, and hydroxides.
3. Additional material soluble in hot dilute hydrochloric acid may consist of carbonates, phosphates, some metal oxides such as magnesium, iron, lead, and other basic compounds.
4. Loss on ignition of the residue should be largely free carbon and carbon compounds. Examples are soot, coal, dust, lint from textiles, and dust from wood and other cellulosic materials. Moisture in terms of water of hydration of inorganic compounds would also be included here.
5. The final inorganic residues may consist of dusts from sand, pumice, quartz, granite, clay, etc.

TABLE III. Analysis of City Dirt *

Component	Pittsburgh	Detroit	Cleveland	Buffalo	St. Louis	Boston	Synthetic
Water soluble (%)	15.4	13.5	15.9	11.4	14.9	15.4	9.8
Ether soluble	10.8	4.9	7.1	6.5	12.8	7.7	5.1
Moisture	—	1.7	3.0	—	—	2.1	8.0
Total carbon	26.4	24.7	24.0	26.9	25.6	28.9	25.8
Ash	53.8	57.8	56.3	52.0	51.2	50.5	40.9
SiO_2 (total)	25.6	25.5	26.4	24.0	24.1	21.4	21.9
Fe_2O_3 (total)	11.6	9.9	11.1	9.5	9.4	11.1	7.5
CaO (total)	6.2	8.4	7.7	6.9	7.4	6.4	4.8
MgO (total)	1.7	2.0	1.7	2.0	1.6	1.7	1.9
CaO (Water soluble)	0.3	0.4	0.7	0.3	0.4	0.7	0.3
MgO (Water soluble)	0.1	0.2	0.2	0.2	0.2	0.2	0.2
N (Water soluble)	—	1.6	—	—	—	2.1	1.3
*p*H (10% slurry)	7.0	7.3	6.7	7.2	7.0	7.3	8.9
Carbon black equivalent (%)	0.8	0.6	0.55	0.5	0.5	0.6	1.2

* Source: *Journal of the American Oil Chemists' Society* [98].

TABLE IV. Synthetic Soil Composition Based on the Analysis of Street Dirt *

Component	%
Humus	35.
Cement	15.
Silica	15.
Clay	15.
Sodium chloride	5.
Gelatin	3.5
Carbon black	1.5
Iron oxide	0.25
Stearic acid	1.6
Oleic acid	1.6
Palm oil fatty acids	3.0
Lanolin	1.0
n-Octadecane	1.0
l-Octadecane	1.0
Lauryl alcohol	0.5

* Source: *Journal of the American Oil Chemists' Society* [98].

TABLE V. Calculated Composition of Typical Carpet Dirt *

Component	%
Moisture	3
Sand, clay, quartz, feldspar	45
Limestone, dolomite	5
Gypsum, apatite	5
Animal fibers	12
Cellulosic materials	12
Resins, gums, starch, etc.	10
Fats, oils, rubber, tar	6
Undetermined	2

* Courtesy: The Hoover Company [112].

collected from a filter in an industrial area was as high as 22%, as shown in Table II.

The Solid Component

The solid fraction of fabric dirt is at least as complex a mixture as the solvent extractable portion. It is made up of an endless variety of organic and inorganic substances—physical and chemical breakdown products of animal, vegetable, and mineral origin. It is present in the form of very small, often microscopic particles, although film-forming substances such as starch or albumen are not uncommon. These latter, together with certain food and body residues, are often associated with localized spotting, while stains are frequently the result of single compounds such as ink, iodine, iron salts, or dyestuffs, rather than complex mixtures. Water-soluble salts from food and perspiration are also found in the dirt present in clothing and household textiles. However, it is not the occasional spotting and staining that presents the most difficult problem to the consumer; the greatest source of trouble is from the finely divided particles and the oils that help to bind them. It is these which cause the overall dulling and discoloration that builds up regardless of frequent mechanical cleaning operations.

Table III shows an analysis of street dirt collected from several large cities over a wide geographical area [98]. Also included for comparison is an analysis of a synthetic soil, the original composition of which is shown in Table IV. This latter was made up to resemble chemically the solids found in city dirt and the oily components of fabric soil as determined by Brown [16]. It is interesting to note the high proportion of inorganic material and the striking similarity between widely separated areas, an observation also reported by Masland [78] and Studer [112] from their studies of carpet dirt.

In view of the widespread use of carbon in various forms as a synthetic soil, it is surprising to note the low carbon black equivalent of these samples, estimated from their comparative coloring value.

Table V gives the composition of a typical carpet dirt derived from the analysis of various samples taken from vacuum cleaners in different parts of the country. This again shows a high content of inorganic materials.

Using x-ray patterns and solvent extraction, it has been shown conclusively [112] that finely divided silica and various oily substances are at least two of the components present in the nonremoved as well as in the mechanically removable portion of the dirt present in naturally soiled carpets.

The results of a third source of information [49], air filtration, are given in Table II. This shows the accumulation of air-borne dirt collected from an installation used to purify the air entering an industrial plant. The material taken from the dry filter is probably more representative since the washed filter may also contain salts deposited from hard water. Again, for atmospheric dust, a relatively high inorganic content can be noted.

Particle Size

Although the chemical composition of fabric soil is an interesting and valuable study, the physical structure of dirt—particle size, size distribution, and geometric shape—may be of even greater significance. Considering its importance, it is surprising that so little information is available on the subject, in spite of the great amount of work that has gone into the development of artificial soils and methods of application for detergency evaluation. Until quite recently this factor has been largely neglected.

From everyday experience it is obvious that the dirt accumulated by textiles includes particles of a wide range of size and shape, which vary from the microscopic materials causing discoloration on shirt collars and cuffs to the readily visible grains of sand, fibrous lint, and hair found on rugs and carpets. The coarse particles visible to the naked eye present little difficulty since they are easily removed in the majority of cases by simple mechanical means. The smaller particles, however, are progressively more difficult to contend with as their size decreases. The materials in the microscopic and submicroscopic range present the greatest problem.

It is difficult to appreciate the great variation possible in the microscopic size range. For instance, visible units still small enough to be called dirt may run from 1000 down to 50 μ; while the microscopic range includes those from 50 to as low as 0.01 μ, a difference of 1 to 5000 as compared with only 1 to 20 for the visible particles. Even those in the upper limits of microscopic size cause little trouble in removal. The greatest difficulty is found in the range below 5 μ. Information is lacking on the particle-size distribution of fabric dirt within this wide range.

As in the case of chemical composition, it should be kept in mind that little is known about the particle-size distribution of the dirt contained in a naturally soiled fabric, and even less about that portion still remaining on the fiber following a mechanical cleaning operation. Most information has been obtained through the study of artificial soils or from an accumulation of dirt only after its removal from the fabric, in which case size distribution can have been altered by either agglomeration or dispersion of particles during removal.

Dust is a common source of fabric soil. The Atomic Energy Commission's *Handbook on Aerosols* [10] gives the following information about the size of air-borne particles:

> An aerosol is an assemblage of small particles, solid or liquid, suspended in air. By small particles is meant a particle with a radius less than about 50 microns. The usual range of particles radii in aerosols is from 0.1 to 10 microns, although particles as small as 0.01 micron may be encountered. The range of particle sizes in the various types of aerosols is considerable. Dust may range from fine particles of 0.1 micron radius or less which produce haze, to sandstorms having large particles beyond the range considered to be aerosols. Smoke is often composed of extremely fine primary particles which have coagulated to form groups. Carbon smoke is composed of small primaries of about 0.01 micron radius which coagulate into long irregular filaments that may reach several microns in length.

Table VI lists the particle-size distribution of street dirt similar to that shown in Tables III and IV. It is interesting to note that more than 50% falls in the range below 5 μ, for which there is no breakdown. An average sample from 14 cities in the United States showed the mass median diameter of atmospheric dust particles to be close to 1 μ [53].

Another source, a sample of vacuum cleaner dirt removed from carpets, ranged in size from 0.3 to 35 μ, while the soil retained by the pile fibers was mostly between 0.2 to 4 μ [78]. Particles from air-borne dust, sand, and metal filings found in used automotive lubricating oil ranged from 2 to 40 μ,

TABLE VI. Particle-Size Distribution of Street Dirt *

Range (μ)	Natural Dirt (%)	Synthetic Dirt (%)
0–4	53	68
4–8	8	16
8–12	7	8
12–16	8	4
16–20	7	2
20	17	2

* Source: *Journal of the American Oil Chemists' Society* [98].

while the organic contaminant particles (carbonaceous soot, macromolecular aldehydes, ketones, acids, and resins) were in the range from less than 0.5 up to 5 μ in diameter [94].

Recent work [22] furnishes strong evidence of the great importance of particle size in soil retention. Figure 1 demonstrates the effect of primary particle size of carbon black on the reflectance of soiled cotton fibers. Using pigments ranging in average size from 13 to 120 mμ (0.013 to 0.12 μ), it shows that the reflectance of a soiled and rinsed fiber pad rises rapidly with increasing soil particle size up to about 50 mμ. The sharp break at this point, followed by the much lower rate of increase in reflectance with the larger particles, is believed to be quite significant.

When cotton fibers become soiled with these extremely small particles, their removal by any means short of destruction of the fiber is virtually impossible. This indicates that carbon particles in this low size range do not make up a large part of the natural dirt encountered in ordinary use since, if they did, soil removal even by severe laundering would be a more serious problem than it actually is. The concentration of particles less than 0.1 μ in naturally occurring dirt is probably so low as to contribute insignificantly to fabric soiling under most conditions [85].

It is important to obtain a mental picture of the size relationships of these extremely small particles in comparison to fibers. Four hundred of them, 50 mμ in size, would fit side by side across the diameter of the average cotton fiber, which in itself is hardly visible without magnification. Fifty millimicrons, 500 Å, is not far removed from the size range of some of the larger organic molecules. Such objects, of course, are far outside the resolving power (about 0.2 μ, 200 mμ) of optical systems. Until the development of the electron microscope with a resolving power of 0.002 μ, such particles and smaller ones

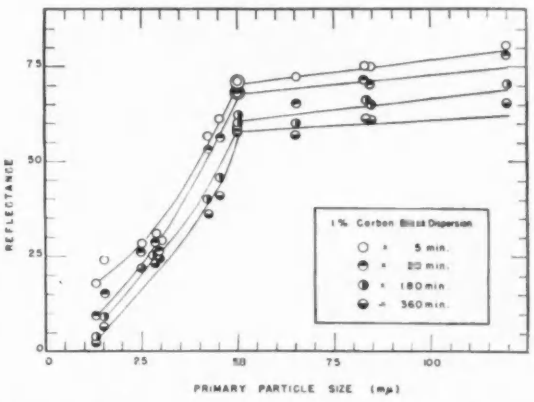


Fig. 1. Effect of primary particle size on rate of change in reflectance.

could not be individually studied. Prior to this, measurement depended on some property of the dispersed system such as viscosity, sedimentation, or light scattering. While these methods are useful for determining the average size of particle groups independent of the fabric, they are not applicable to the measurement of dirt particles in situ.

Through electron microscopy and other methods, considerable information is available [20] concerning the individual size, shape, and size distribution of a wide variety of carbon blacks, many of which have been used as artificial soils. Such information should be taken into consideration whenever these pigments are used for artificial soiling in detergency studies.

Figure 2 shows four different carbon blacks whose mean particle diameters range from 274 down to 28 mμ. Since all are at the same magnification, their sizes may be compared directly. The average size of the largest of these (0.274 μ) is just within the visible range of the optical microscope. It is interesting to note the chainlike structure characteristic of these particles.

2. Mechanisms of Soiling

The questions concerning the nature of the forces involved in the fiber-soil complex are fundamental to an understanding of its formation and stability. The forces of impingement act to bring the two components together; while the forces of retention cause them to stay together as a more or less stable unit once they are in contact. In most instances, the two are separate and distinct mechanisms.

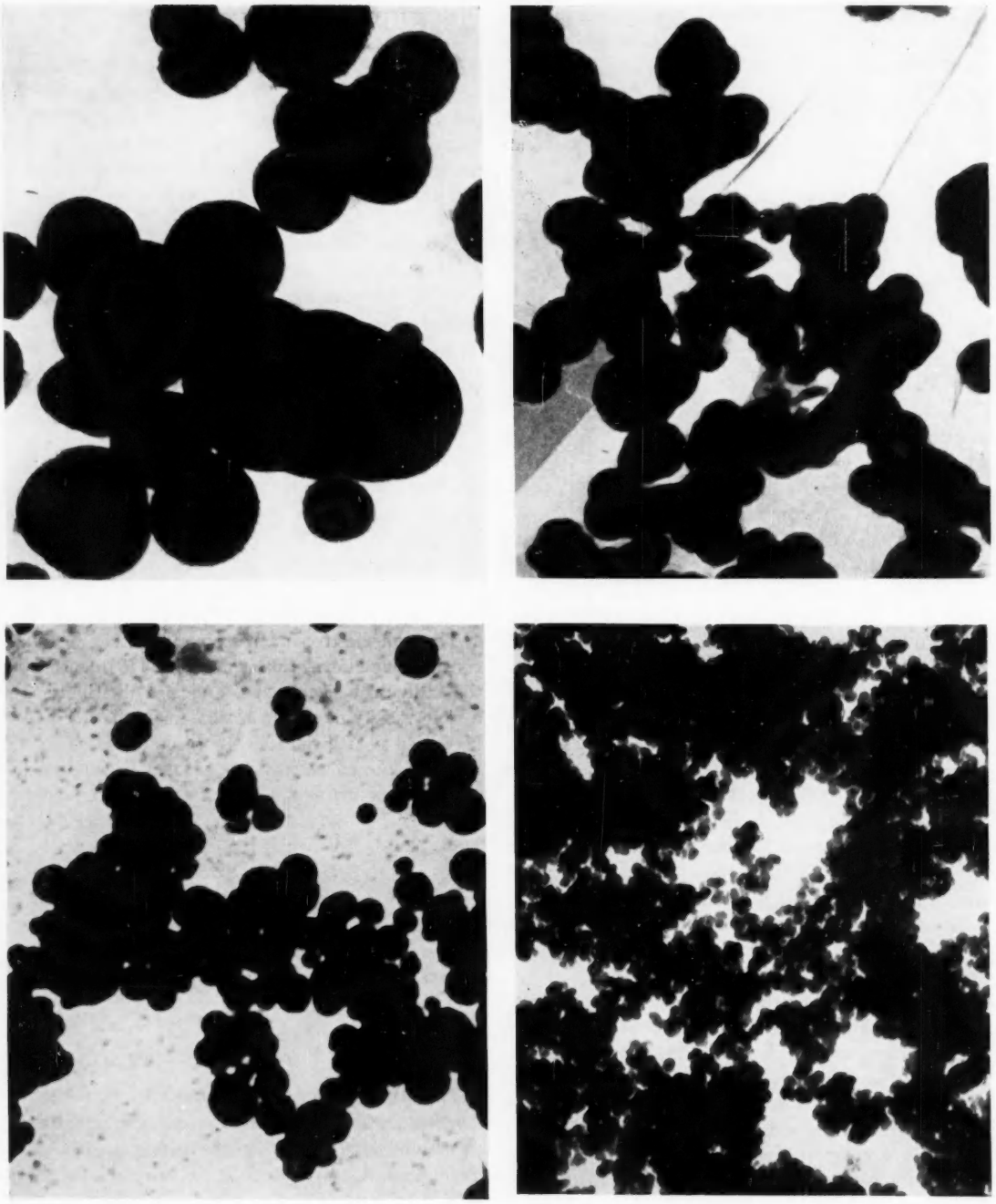


Fig. 2. Electron micrographs of carbon black particles ×50,000. *A*, Thermax; mean particle diameter 274 mμ. *B*, Lamp black; mean particle diameter 97 mμ. *C*, P-33; mean particle diameter 74 mμ. *D*, Micronex; mean particle diameter 28 mμ.

The Forces of Impingement

Fabric and dirt come together either by direct contact with a soiled surface or by contact with air-borne or liquid-borne substances.

Through direct contact, simple mechanical forces act to transfer oily and/or solid material directly from a soiled surface to the surfaces and spaces available within a fabric. It is an everyday occurrence to soil clothing by contact with an object or to transfer oil and grease to a carpet from the soles of shoes.

Fabrics may also collect dust and dirt from the air or pick up dissolved or suspended substances from liquids with which they come into contact. The transportation of soil into fabric structures by fluids involves the principles of filtration. Textile materials act as filters when fluids pass through them by virtue of either the motion of the fluid in relation to the fabric or the movement of the fabric through the fluid. A good source of information on the merchanisms of particle motion in aerosols and the theories of air filtration can be found in handbooks published by the Atomic Energy Commission [10, 11]. A comprehensive review of this and its relationship to fabric soiling is presented in an article published by a committee of the AATCC [85].

The motion of individual dust particles in the air is influenced by several factors. Brownian movement, resulting from molecular bombardment, causes the random oscillations of very fine particles and their diffusion to solid surfaces or collision and coagulation into larger particles. In filtration, removal of particles by diffusion is most effective in the size range of 0.1 μ and less, and is promoted by low air velocities through the filter.

Thermal forces cause movement of particles toward objects colder than their surroundings, while convection currents set up by temperature differences or by mechanical forces cause movement of particles, bringing them into contact with surfaces and each other. Static electrification results in the precipitation of particles onto charged surfaces, and may, under certain circumstances, be a highly important factor in their removal by a filter. Gravity causes particles to settle out of the air and and deposit onto any surface having a horizontal component—the larger the particle, the greater the rate of settling. Particles in the range of 0.3 μ and larger tend to settle out as they pass through a filter. Inertial effects are important for heavier particles, 1 μ and more, since, when a streamline bends sharply around a fiber, a heavy particle at high velocity tends to continue in a straight line and collide with the fiber rather than follow the sudden bending.

Removal of particles between 0.1 and 1 μ is the most difficult in air filtration since it depends largely on a chance collision between a particle and fiber of the filter. Because of greater surface area and more sharply bent streamlining around the smaller diameter, fine fibers are always more efficient than coarser ones in removing particles in all size ranges. However, for practical purposes, a filter must be sufficiently permeable to allow passage of large volumes of air. It must not be dependent on the sieving action alone of fine, closely packed fibers that quickly plug up with dirt and restrict the flow. Since the fibers in most cotton fabrics are more closely packed together than in the ordinary filter medium, the chances of collision or direct interception of particles in an air stream are greatly increased.

The mechanisms of particle motion and filtration apply in varying degrees to all fluids, liquid as well as gaseous. In a similar manner to that for air-borne particles, solids suspended in water, oil, or other liquid media may come into contact with the fibers of a fabric.

Liquid-borne substances are frequently the cause of staining and spotting. Splashing of muddy water on clothing, for example, or tracking of dirt and water on carpets in wet weather is a common experience. Dirt originally picked up from the air or by direct contact with a soiled surface may be transported deeper into the fabric by later contact with liquids. Whenever fabrics are washed or dry cleaned, they are subject to the form of liquid soiling known as redeposition.

The swelling of the fiber by polar liquids and the subsequent deswelling upon drying may alter the position of soil particles in relation to the fiber surface. Also, polar liquids can transport dissolved substances through the fiber wall into the internal structure of the fiber. Deposition of dissolved or suspended substances by evaporation of the solvent is a mechanism of impingement present only in the case of liquids. The large force exerted by surface tension as the liquid evaporates may bring small particles into very close contact with fiber surfaces or with each other and result in strong adhesional bonds [24]. It has been calculated that a collapsing force as high as 2000 psi is exerted on a column of 0.02 μ in diameter by an evaporating film of water [4].

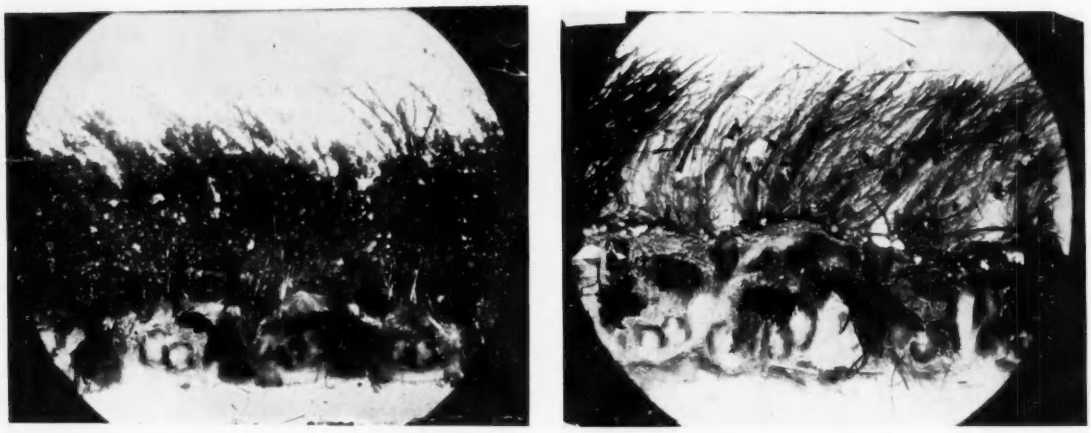


Fig. 3. *A*, Photomicrograph of a cross section of a very dirty rug showing quantities of dirt that can collect in the pile. *B*, Same rug after mechanical cleaning.

Table VII lists seven possible mechanisms of impingement together with a particle-size classification for air-borne dusts.

The Forces of Retention

Snell [109] states that dirt is retained in a fabric by mechanical and electrostatic forces, and by oil bonding. A similar conclusion was reached by the New York Section, AATCC [85] which mentions that ". . . particles may adhere to fibers by mechanical forces or occlusion in pits and crevices on fiber surfaces, by 'oil' bonding, and possibly by electrical forces."

Compton and Hart [22] list the following mechanisms which may be responsible for retention:

1. Macro-occlusion, or entrapment of particles in the intra-yarn or inter-yarn spaces.
2. Micro-occlusion, or entrapment of particles in the irregularities of the fiber surface.
3. Sorption of materials by van der Waals or coulombic forces at the surface or within pores and crevices.

TABLE VII. Mechanisms of Impingement

Contact Mechanism	Particle Size Classification (Microns) (Air-Borne Materials)
1. Direct transfer from soiled surface	—
2. Diffusion	<0.1
3. Electrostatic attraction	All
4. Gravity settling	>0.3
5. Inertial effect	>1
6. Chance collision	0.1 to 1
7. Deposition by solvent evaporation	—

Schwartz [103] also mentions three types of forces holding together the fiber-soil complex: "(*a*) Pure interfacial, adhesional forces, i.e., van der Waals forces of intermolecular attraction . . .; (*b*) Coulombic, or electrostatic forces . . .; and (*c*) Purely mechanical forces of juxtaposition and entanglement. . . ."

Authorities on the subject agree as to the type bonds involved in the adhesion of dirt to fabrics; uncertainty arises only in respect to their relative importance.

Forces of retention are of two distinct types: (1) those dependent on some form of energy bond; and (2) mechanical entrapment.

Mechanical entrapment is undoubtedly responsible for the largest *weight* of dirt accumulated by most textile materials; it may also be the cause of some of the most tenaciously held soil. To appreciate fully its importance, an understanding of fabric, yarn, and fiber structure is necessary.

Soil may be contained within a fabric structure in four general areas—in the relatively large spaces between the yarns, in the smaller system of pores between the fibers of a yarn, in the tiny angles formed by single fibers, and in or on the microscopic fiber surface itself. Yarn and fabric construction determine the geometry of the first two areas, while fiber morphology governs the latter two.

Weave and yarn constructions of cotton fabrics vary widely. At one extreme is the open porous condition of a pile fabric; and at the other, the dense,

compact surface of ducks or broadcloths. The tightly woven structure of the latter prevents the entrance of all but the smallest particles; while the open surface of a rug pile offers little hindrance to the penetration of large quantities of coarse-grained dirt. On the other hand, the rug pile also offers less resistance to the removal of dirt by mechanical means. The cross sections of the dirty and the cleaned carpets shown in Figures 3*A* and 3*B* illustrate effectively the quantities of dirt that can become embedded in such open structures as well as the ease with which most of it can be removed by modern cleaning devices. In both cases it is the oily or fluid soils and the fine particle-size dirt, small enough to come into intimate contact with the fiber surface, that present a problem of removal.

Theoretically, the fiber having the minimum of surface area per unit of volume will have the maximum resistance to mechanical soil entrapment, i.e., a fiber having a large diameter, circular cross section, and a perfectly smooth surface.

Masland [78] in 1939 published the results of a study of soil retention of wool and viscose fibers used in carpets. Dirt particles were often found trapped in the gross irregularities of the fiber surface—in wool, at the edges of the scales (Figure 4); in ordinary viscose, in the channels running lengthwise of the fiber; and in cotton, in the depressions formed by the convolutions (Figure 5).

He concluded: (1) that lowest soil retention is encountered in fibers of large diameter (above 27 μ) and smooth, circular, cross-sectional outline free from channels; (2) that fiber length is not a factor in soil retention; and (3) that the origin of the fiber—animal, vegetable, or synthetic, is immaterial. He stated that dust retention is a function of the external areas of the fiber, and that, for fibers of

Fig. 4. Electron micrograph of naturally soiled wool fiber showing concentration of particles at the scale edges.

Fig. 5. Photomicrograph of soiled cotton fiber showing tendency of particles to concentrate in fiber folds.

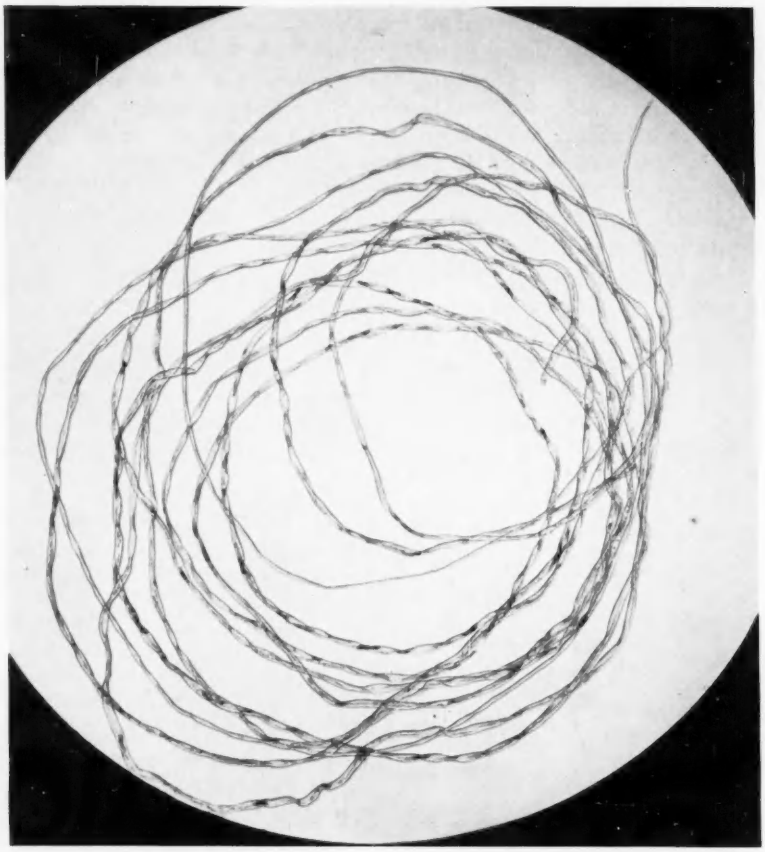


Fig. 6. Photomicrograph of single cotton fiber, ×70.

smooth, approximately circular cross sections, soil retention per gram of fiber is proportional to the total surface area.

Most textile fibers do not possess the ideal shape for soil resistance; in fact, if they did, they would be less adaptable for textile uses in other respects. Of the natural fibers, coarse wool and mohair come closest and are known for their good cleanability. Synthetic fibers can also be produced to closely approximate these conditions, as in the case of the special coarse-denier carpet rayons now on the market [79, 125].

The cotton fiber in its natural state does not fit the ideal pattern. Its diameter is small, averaging 16 to 21 μ; its cross section is in the form of a collapsed and twisted hollow tube; and its surface is rough and irregular. Figure 6, showing the full length of a single cotton hair, and Figure 7, a portion at higher magnification, demonstate the possibilities for dirt entrapment in the ribbonlike convolutions.

Although little experimental evidence is yet available, it is likely that cottons may differ somewhat in their soiling characteristics because of the influence of variety and maturity on shape. Figure 8*A* shows a coarse, mature variety, while 8*B* is a much finer type. It can be seen that the coarse, rounder fibers of the former contain fewer gross irregularities in which soil particles may lodge.

The fine surface structure of the cotton fiber is also an important area for soil entrapment. Much of the surface detail as well as some of the most tenaciously held dirt particles are beyond the range of the ordinary light microscope. The electron microscope, however, because of its great depth of field, furnishes an excellent means of studying the size, shape, and location of the dirt as well as the fine details of the fiber surface to which it is attached. The crevices and ridges shown in Figure 9*A–F* are typical of the fine surface structure of cotton. Figure 10*A–H* shows the surfaces of several other fibers as a means of comparison.

Figure 11 is a schematic diagram of the structural layers of the cotton fiber, of which the primary wall, or outermost layer, is of particular interest since it

is ordinarily the only surface that comes into contact with the dirt. In its thickness of between 0.1 and 0.2 μ is contained most of the noncellulosic materials present in the fiber. Figure 12 shows the primary wall peeling from the fiber's surface after beating in water; while Figure 13 shows a series of electron micrographs of sections of the primary wall after removal by mechanical action from the underlying body of the fiber. Figure 13*A* shows a specimen from an untreated fiber, while 13*B* and *C* were taken from fibers treated to simulate ordinary cotton-finishing operations. Figure 13*D* is a section highly purified to show the networklike structure of fibrils visible only after complete removal of the waxes and pectins, a condition not ordinarily encountered in commercial practice. Figure 14 is an electron micrograph of the fibrillar structure present in the secondary wall of the cotton fiber. Such subsurface layers may be exposed when the primary wall is ruptured through wear or fiber breakage. Evidence that the primary wall may undergo extensive damage during kiering has also been reported [60].

Particles which can be mechanically trapped by such minute irregularities must necessarily be of extremely small dimension. For instance, the fibrils visible in the network structure shown in Figure 13*D* have been estimated [114] to be of the order of 100 to 400 Å in diameter (0.01 to 0.04 μ). From this it can be seen that the crevices and depressions between are roughly of the same or slightly larger size, possibly in the range of 500 to 600 Å. As mentioned

Fig. 7. Photomicrograph of cotton fibers showing convolutions, ×550.

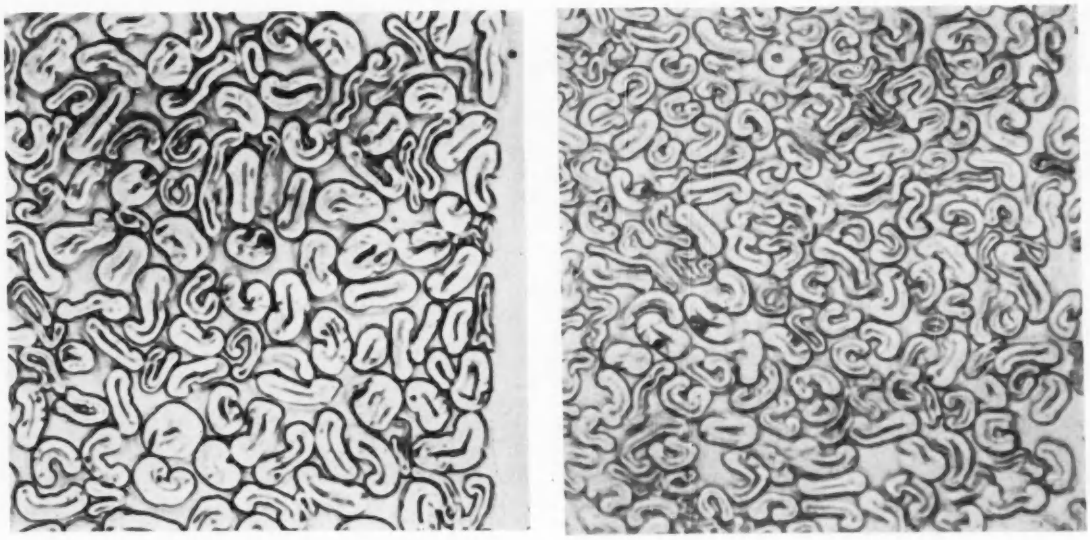


Fig. 8. Cotton fiber cross sections, ×500. *A*, Coarse mature variety. *B*, Fine immature variety.

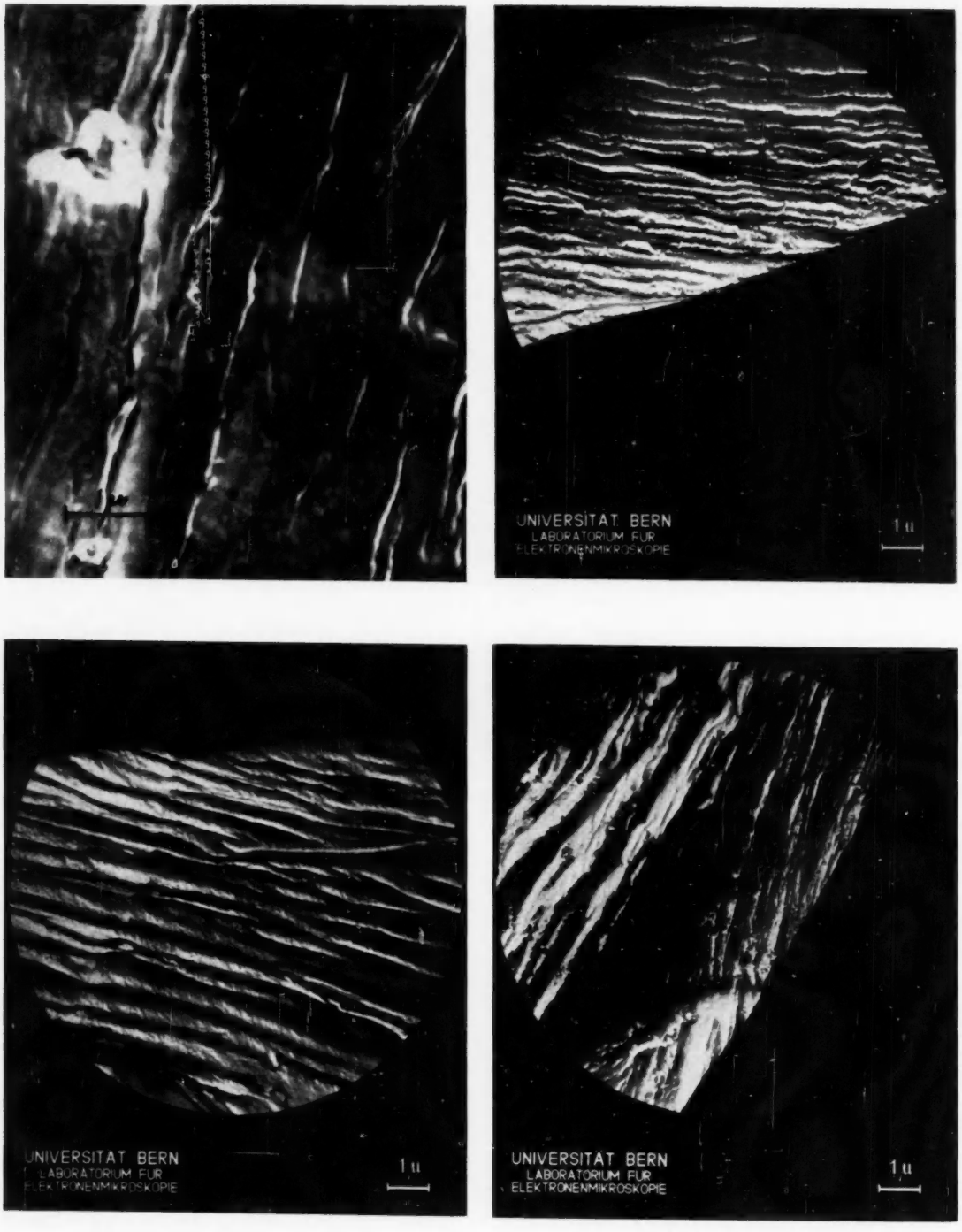
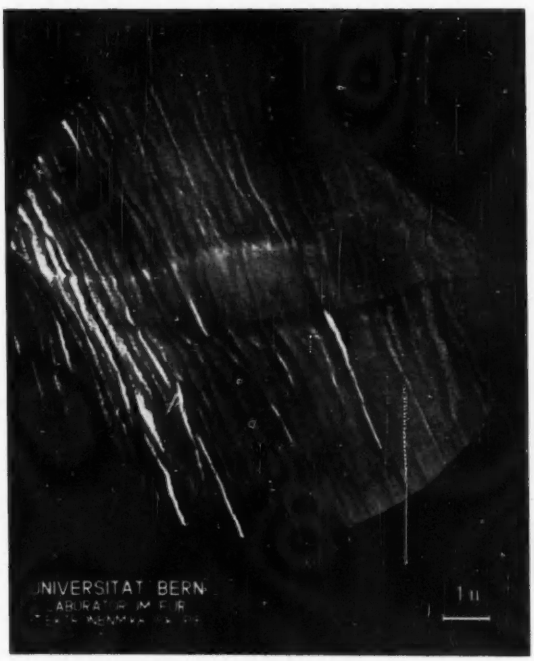


Fig. 9. Electron micrographs showing



the surface of the cotton fiber.

earlier, naturally occurring dirt particles in this size range are probably rare.

However, carbon black particles of around 500 Å and smaller are known to soil cotton severely. In connection with this, the work of Compton and Hart [22] is of particular interest in respect to the mechanical theories of soil retention. See Figure 1. They state:

> By use of the electron micrograph, numerous crevices of 50 millimicrons in diameter and less may be seen on the surface of the cotton fiber, whereas larger ones are rare. It would be expected that the number of soil particles to the fiber surface would increase very rapidly as the soil primary particle size decreases below the value of 50 millimicrons. This was found to be the case.

Experiments were carried out on chopped cotton fibers in aqueous dispersions with carbon blacks in a wide range of particle size, as soil. They concluded that, in the *absence of grease and oil*, particles were bound to the cotton fiber surface primarily by geometric bonds or microocclusion rather than by energy bonds or sorptive forces. These latter, although not excluded from a role in the *formation* of the complex, were considered to be of minor importance once the complex was formed. It was suggested that the geometric relationships between the size and the shape of the soil particles and the fiber surface irregularities and the probability of the close approach of the two are the main factors in both formation and stability of the fiber-soil complex. Such geometric bonds are in some cases so strong that only disruption of the fiber can release the soil particle. The difference between the effect of mechanical energy and heat energy on stability of the complex was felt to be quite significant. An input of 1000 cal of mechanical energy in the form of stirring significantly decreased the number of attached soil particles; whereas a 25,000 caloric heat-content differential had no effect. It is claimed that, while the hypothesis of geometric bonding predicts such an effect, no form of energy bond can explain it. Table VIII lists other factors which led to the above conclusions.

Other forces of adhesion all involve some form of energy bond. They include a wide range of physicochemical phenomena varying all the way from the very strong primary chemical valence bonds, involving ions and orbital electrons, to the relatively weak van der Waals forces which are the basis of attraction between the molecules of a gas. To differentiate from mechanical adhesion, forces dependent on

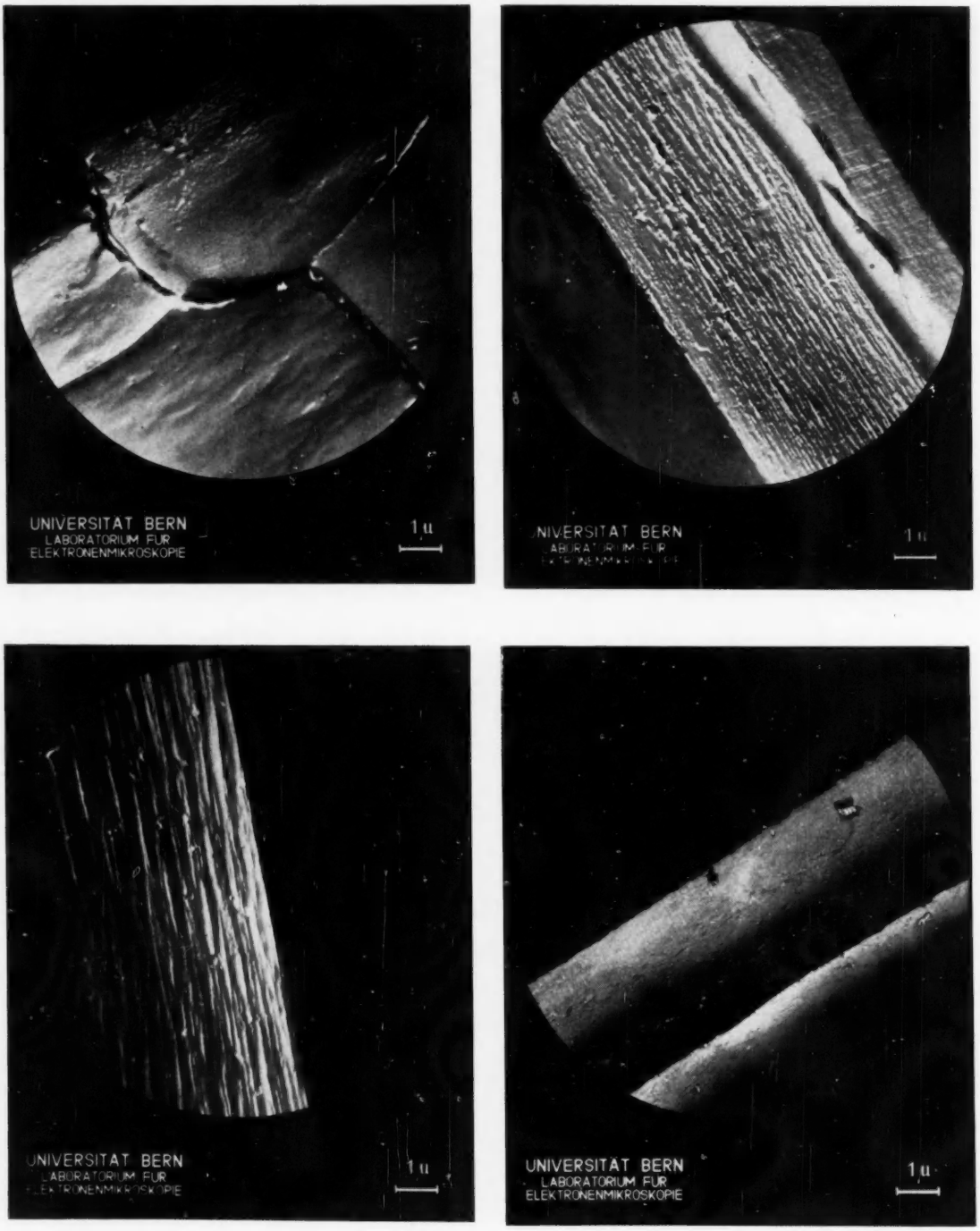


Fig. 10. Electron micrographs of fiber surfaces, ×7000. *A*, Wool; *B*, silk; *C*, nylon; *D*, viscose rayon.

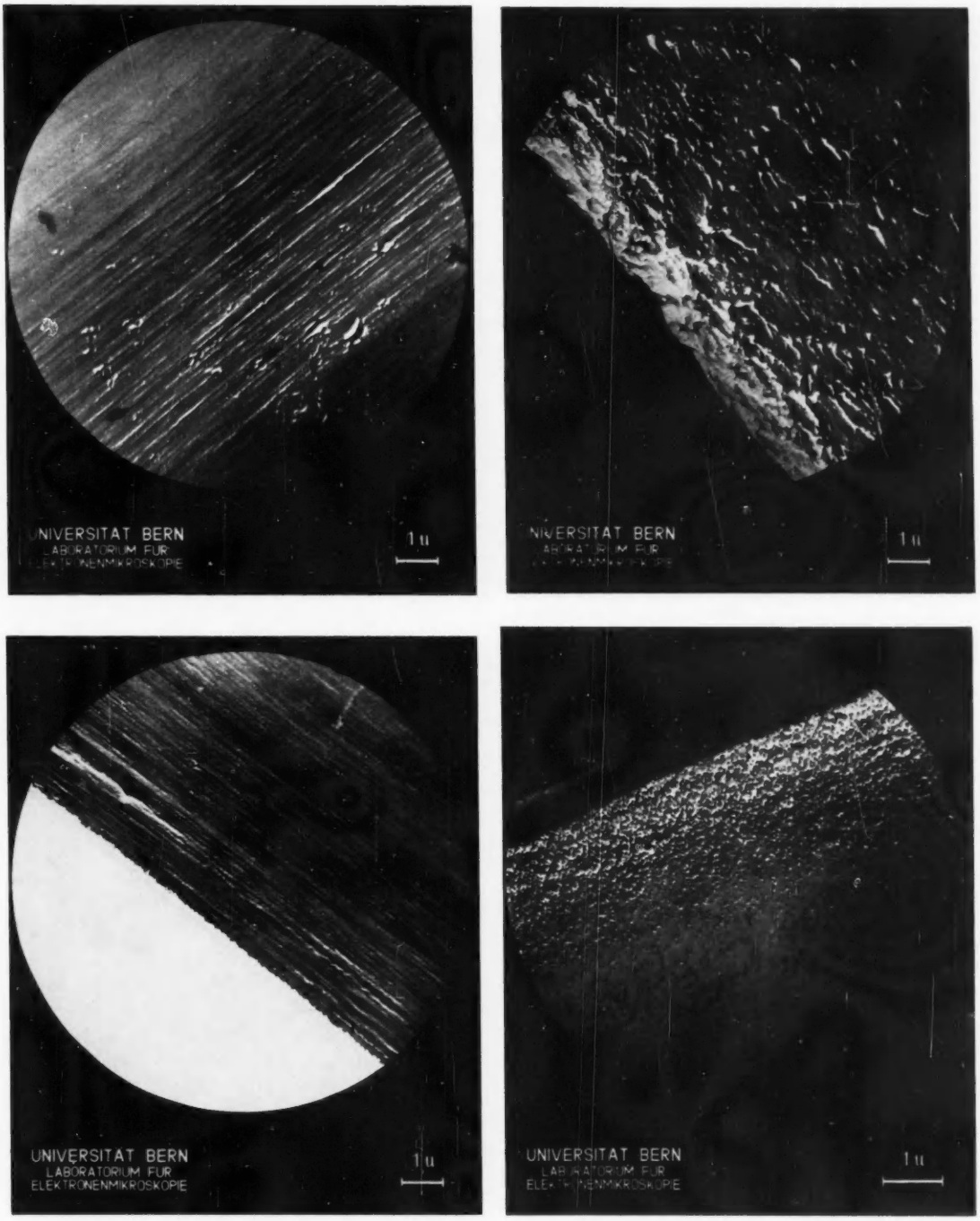


Fig. 10. Electron micrographs of fiber surfaces, ×7000. *E*, Vinyon; *F*, Pe Ce fiber; *G*, polyacrylic fiber; *H*, Terylene fiber treated with caustic soda.

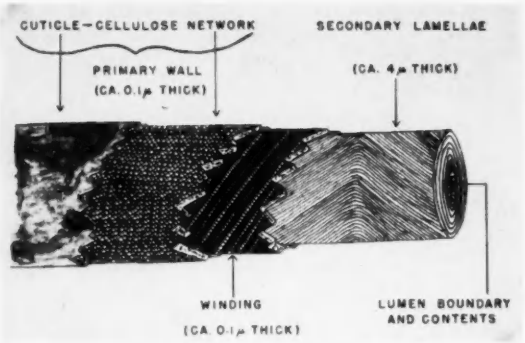


Fig. 11. Schematic diagram of cotton fiber morphology.

some form of energy bond have been called "specific adhesion." They are also termed "sorptive forces" since they involve only surface reactions.

Since ordinary soiling is believed to be a purely surface phenomenon, for cotton at least, the adhesional forces existing at the interface between solid-solid and solid-liquid systems are fundamental. Most of the information in this field has been built up through studies of surface films involving liquids in one or both phases of the system. Because of experimental difficulties, knowledge of the adhesional forces existing between solids is much more limited, and thus some confusion as to the exact roll played by sorptive forces in the attachment of solid soil particles to fibers can be expected.

Chemical forces—Staining. In ordinary usage, the words "soiling" and "staining" are employed loosely and sometimes interchangeably. The term "stain" has always implied a degree of permanency not associated with "soil" or "dirt." While the latter can be removed by ordinary cleaning or laundering, it is usually necessary to employ special means to remove a stain. A means of distinguishing between the two in a technical sense would be useful. The answer may lie in whether or not the contaminating material has penetrated beyond the surface of the fiber.

Experimental evidence [12, 39, 50, 51, 104, 123] indicates that cotton contains a system of openings or pores leading from the outer surface through the primary wall to the inner structure of the secondary layers. In the dry fiber these openings are extremely minute, and the internal areas are available to only the smallest molecules. When swollen by water or NaOH solutions, much larger molecules have access to the amorphous or less highly oriented areas within the fiber structure. Even with swollen cotton, however, only substances in molecular solution can enter and then only if they fall within a certain minimum size range.

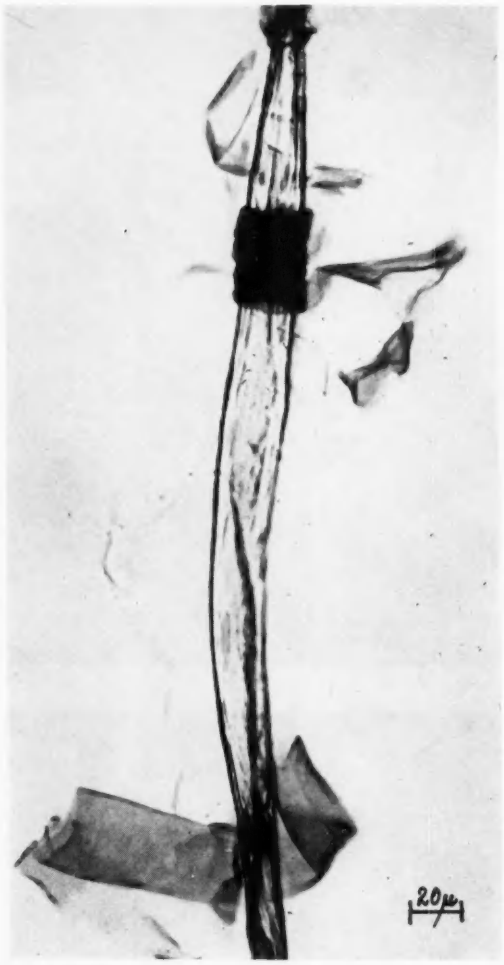


Fig. 12. Photomicrograph of primary wall peeling from fiber surface after beating in water in Waring Blendor.

Useful definitions for soiling and staining can be formulated by distinguishing between those substances that penetrate and those that do not penetrate the fiber surface. While even the finest colloidal dirt particles and the large, nonpolar molecules of oils and greases are wholly restricted to the fiber surface, most water-soluble inorganic salts and many soluble organic molecules such as dyestuffs can penetrate to the internal areas of the swollen fiber. As recognized in current advertising for stain-resistant fabric finishes, stains are usually the result of contact with such aqueous solutions. Therefore, except for the inevitable borderline cases, soiling can be considered a surface condition, and staining a function of internal areas.

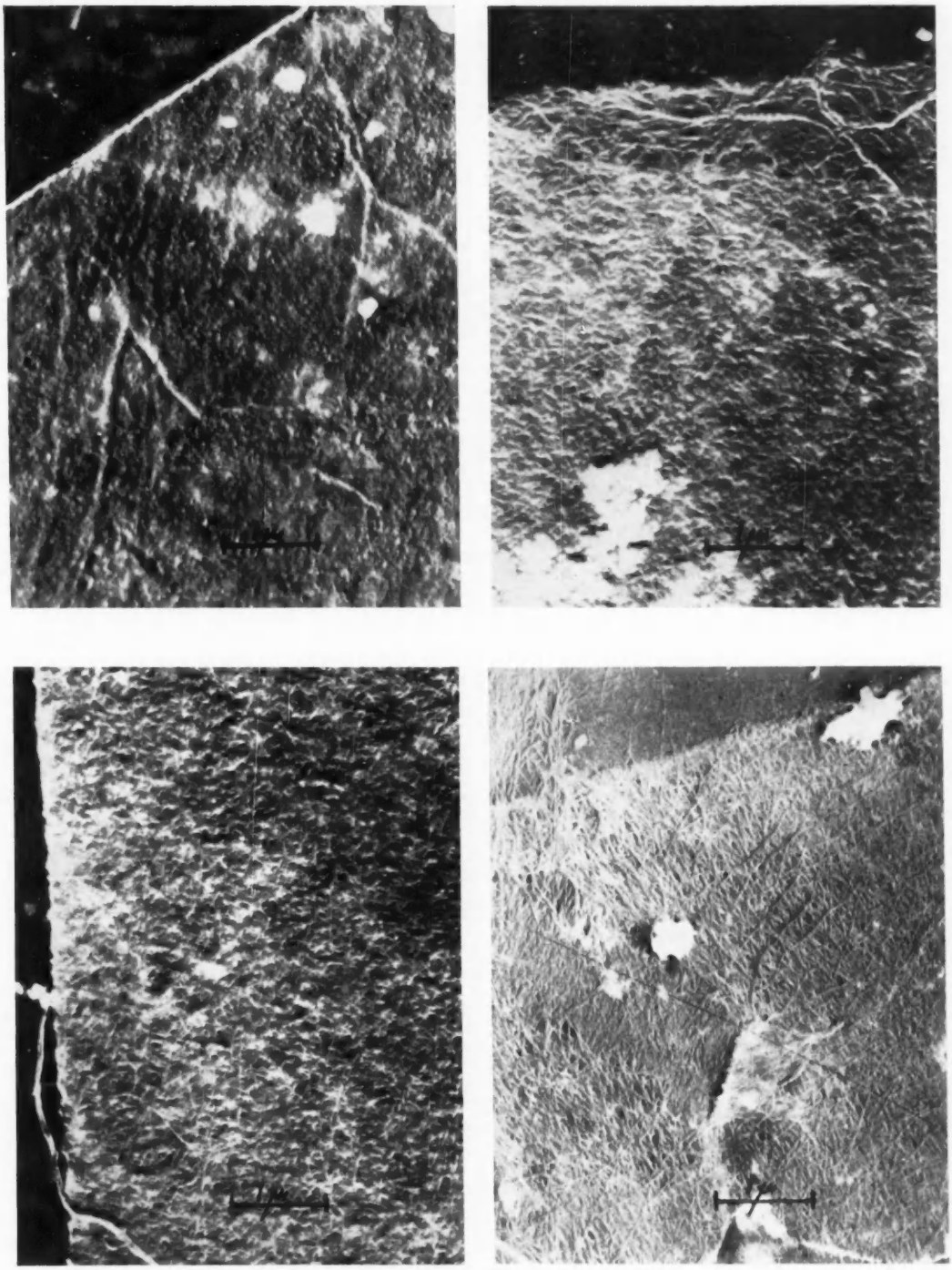


Fig. 13. Electron micrographs of specimens of primary walls: *A*, prior to removal of noncellulosic material; *B*, after kiering in 2% sodium hydroxide with a wetting agent; *C*, bleached for 1 hr room temperature with Clorox after an 8-hr kier at 15 lb pressure in 3.6% sodium hydroxide; *D*, subjected to 6 *N* HCl at 100°C for 6 hr after purification by extraction with hot ethyl alcohol followed by monoethanolamine (note the two-directional system of fibril arrangement).

Fig. 14. Electron micrograph of one of the inner layers of the cotton fiber (secondary wall cellulose), ×17,000.

Primary chemical bonds, as they are normally understood, play no part in the soiling of cotton, and only very rarely can they be considered in relation to staining. The mechanisms by which stain-forming substances are attached to cotton are similar to those involved in dyeing—a subject well covered by the literature in that field. Secondary valence forces, hydrogen bonding, as well as other forces of electrostatic and molecular attraction, and even mechanical occlusion in the case of insoluble particles formed within the fiber, are believed to be of greatest significance, at least in the case of cellulosic fibers. Thus, staining is basically like soiling, involving both mechanical and sorptive forces, but on a molecular scale and at internal rather than only external surfaces.

Electrostatic forces may be involved in the retention as well as in the attraction of dirt. The AEC *Handbook on Aerosols* [10] mentions that small particles adhere to any surface they contact because of van der Waals forces, and that when electrostatic charges are present, the force of adhesion may be increased. Snell [109] states that, in the absence of oil bonding, electrostatic forces constitute one of the important factors in the direct adhesion of soil to fabrics. While electrostatic attraction is well recognized as a common mechanism of impingement, its effectiveness as one of the components of the adhesional force present in the fiber-soil complex is not well established.

That large surface charges can be built up on fibers by friction is evident from the precautions that must be taken to guard against this factor in textile processing. For cotton, the static problem is less severe than for most other fibers, particularly the newer synthetics with low moisture capacity. In fact, the static charges that can be produced on plastic surfaces are so large that they have been used for dust removal in filtration [121].

Electrostatic soiling usually results from the charges present on fabric surfaces rather than those on the soil particles, since the concentration of charged particles in the air is low under normal conditions. Although frictional forces can produce dust clouds with charged particles, charges tend to disappear quickly through agglomeration and neutralization of oppositely charged particles [10]. Charged fabric surfaces attract particles that come within range by inducing a charge of opposite sign on the surface of the particle, the force varying inversely as the square of the distance. Dust particles of carbon black, ash, and short wool and rayon fibers are known to be equally attracted by the positive and negative areas found in close proximity to each other on the surface of plastic films [128].

After a thorough review of the literature, an AATCC committee [85] drew the following conclusions concerning the effects of electrostatic forces in textile soiling:

1. Most particles are uncharged and are not drawn to fabrics by virtue of *their own* electrostatic conditions.
2. Frictional forces and other naturally occurring conditions can probably induce static charges of short duration on many soil particles. If these particles come close to a textile before the charges are dissipated, they may be drawn into direct contact, where mechanical forces and oil bonding can come into play.
3. Uncharged particles may be strongly attracted to fabrics which have become charged.
4. The field requires further investigation.

The possibility of reducing soil retention by discharging the static forces that might exist in the pile of a wool carpet has been investigated by means of placing radioactive materials in the nozzle of a vacuum cleaner [47]. No noticeable improvement in soil removal was observed, however. Electrostatic soiling is by no means restricted to air-borne dust. In dry-cleaning processes it is well known that fric-

tional charges built up by flow of the liquid through the fabric attract dirt and lint particles [15, 40]. Electrostatic attraction also plays a large part in the filtration of motor oil [129]. It has been shown that surface-active agents added to cotton and wool fibers of the oil filter increase the removal of organic materials but, it is interesting to note, not of sand and metal particles [94].

Even in aqueous media electric charges of a low order may exist on both fibers and soil particles. Electrophoretic studies show that cellulose as well as many of the soils present in textile materials have weak negative charges [62, 109]. Under certain circumstances, however, finely divided carbon black and iron oxide may be positively charged and thus be attracted to the negatively charged cellulose [42, 109, 110]. From a series of experiments, Utermohlen [116] concluded that treatments which can impart an electric charge and cation-exchange properties to cotton were the ones most effective in reducing soiling.

Without further evidence to the contrary, it may be concluded that electrostatic soiling does not present a serious problem for cotton. Although under certain conditions cotton may accumulate surface charges and thus attract and retain dirt particles, the charges are quickly dissipated under normal atmospheric conditions, after which retention must depend on some other mechanism of adhesion.

Oil bonding. Oil bonding is the term aptly coined to describe the commonplace phenomenon of adhesion of dust and dirt to oily or greasy surfaces. Oils and greases are not alone in this respect, since a surface, whether wet by oil, water, or any other material having the capacity to flow, will exhibit the properties of binding small solid particles. In the case of volatile liquids, however, the adhesion due to wetting disappears upon evaporation.

The adhesive force of oil bonding results from a mutual wetting of the surfaces of fibers and of the soil particles. Wetting is a sorptive mechanism ascribed to van der Waals forces, the basic molecular bonds of attraction that exist between the particles of all matter—gaseous, liquid, or solid. They are said to vary inversely as the sixth power of the distance and are thus effective only over a very short

TABLE VIII. Predicted and Observed Effects of Microocclusion and Sorption on Formation and Stability of Soil-Fiber Complexes *

		Predicted Effect from Sorption		Predicted Effect from Microocclusion		Experimental Result	
		Formation	Stability	Formation	Stability	Formation	Stability
Decreasing particle size of soil		+++	···	+++	···	+++	···
Abrasion of surface	Fine blacks	None	···	None	···	None	···
	Coarse blacks	None	···	++	···	++	···
Effect of detergents	Fine blacks	−−−	−−−	−−	None	−−	None
	Coarse blacks	−−−	−−−	−	None	−	None
Effect of salts and *p*H changes	Fine blacks	++	++	±	None	±	None
	Coarse blacks	++	++	±	None	±	None
Effect of mechanical agitation	Fine blacks	···	None	···	−−	···	−−
	Coarse blacks	···	None	···	−−	···	−−
Effect of fiber pretreatment with detergents	Fine blacks	−−	···	None	···	None	···
	Coarse blacks	−−	···	None	···	None	···
Effect of fiber pretreatment with salts	Fine blacks	++	···	None	···	None	···
	Coarse blacks	++	···	None	···	None	···
Microscopic observations		Distribution of soil uniform		Soil particles concentrated in crevices		Soil particles concentrated in crevices	

− Slight decrease in number of particles bound. + Slight increase in number of particles bound.
−− Marked decrease in number of particles bound. ++ Marked increase in number of particles bound.
−−− Large decrease in number of particles bound. +++ Large increase in number of particles bound.

* Source: *Industrial and Engineering Chemistry* [22].

molecular range. Van der Waals forces are, therefore, relatively unimportant as a means of attraction for dirt particles.

As a mechanism of retention, however, such sorptive forces are important for those soils that can come into close molecular contact with fiber surfaces. In addition to free-flowing liquids, these include the more viscous and semisolid substances that, through pressure or temperature, can be deformed to give an appreciable area of surface contact with the fiber. Pressures exerted by evaporating films of liquid tend to deform the surfaces and bring soil and fiber into closer contact, resulting in increased effectiveness of the sorptive bonds. However, with discrete solid particles of dirt, except for those so small as to approach molecular dimensions and cause staining, it is difficult to see how, in the absence of oily films, van der Waals forces can exert any appreciable force of retention because of the relatively large distances and small areas of contact involved between particle surfaces and fiber surfaces.

From a practical point of view, the importance of oil bonding as a mechanism of solid-soil retention should not be underestimated. Rapid resoiling of rugs when oily residues are left in the pile after cleaning is a problem recognized by the carpet-cleaning industry [70, 71, 73]. New carpets are known to soil more quickly when oils from the manufacturing process are left in the pile, or when processing oils present in jute backing yarns migrate to surface fibers [37, 125, 127]. Current theories of laundering and dry cleaning are based on the premise that a large part of fabric dirt is attached through an oily or greasy layer, and that the function of the detergent or dry-cleaning fluid is to break the fiber-oil and oil-soil bonds and keep the released particles from redepositing until they can be flushed away. Because of their stability in hard water, synthetic detergents provide a means of preventing the greasy deposits of insoluble calcium and magnesium soaps that increase subsequent resoiling.

Whether fiber surfaces are ever completely free from oily films under normal conditions of use is doubtful. Adam [1] points out that a freshly formed solid surface soon becomes coated with a film of greasy material if left unpreotected for only a short time in ordinary air. Such thin, possibly monomolecular layers adhere tenaciously to a surface and are difficult to remove by ordinary methods. Adhesional and frictional characteristics of completely clean surfaces are known to differ radically from those contaminated with thin films. For instance,

TABLE IX. Most Probable Soiling Mechanisms for Various Textiles *

Fabric and Conditions	Mechanism of Impingement	Mechanism of Retention	Normal Service Time for Soiling
1. Light curtains at open window	Interception and inertial effect	Occlusion, oil bonding by finishing agents	Few weeks–few months
2. Light curtains at closed window	Diffusion and deposition	Occlusion, oil bonding by finishing agents	Few weeks–few months
3. Suitings	*a.* Sleeves, seat, and cuffs—direct transfer	Occlusion	*a.* Few days
	b. Top of shoulders—deposition		*b.* Few weeks
	c. Body—direct interception and diffusion		*c.* Few weeks
4. Women's stockings	Interception, direct transfer	Occlusion, oil bonding by skin secretions	Few hours–few days
5. Men's shirts	*a.* Sleeves, collar, and cuffs—direct transfer	*a.* Occlusion, oil bonding by skin secretions	*a.* Few hours
	b. Top of shoulders—deposition	*b.* Occlusion	*b.* Few days
	c. Body—interception or diffusion	*c.* Occlusion	*c.* Few days
6. Carpets and rugs	Direct transfer, deposition	Occlusion, oil bonding	Few months
7. Working clothes	Direct transfer, interception	Occlusion, oil bonding	Few hours–few days
8. Flags	Interception, diffusion, inertial effects	Occlusion	Few weeks–few months
9. Tents	Deposition, direct transfer, interception	Occlusion, oil bonding by fireproof finish if any	Few weeks
10. Gloves	Direct transfer	Occlusion	Few hours–few days

* Source: *American Dyestuff Reporter* [85].

extremely accurate metal gage blocks when completely free of moisture have little tendency to adhere; but even a very thin film of water between the surfaces produces a high degree of adhesion. Adhesion of paint films to metal surfaces is reduced by even monomolecular layers of oil or grease [107]. A further example is shown by the results of an experiment [13] in which the adhesional force between a carefully grease-extracted, untwisted filament yarn and a carefully cleaned glass cylinder was found to be very high. Allowing the glass cylinder to stand in the air for as little as 1 hr after cleaning drastically reduced adhesion, presumably due to contamination of the glass surface.

Fiber surfaces are also subject to atmospheric contamination by oily films; and it is quite certain that the usual dry-cleaning or laundering process does not remove the last traces of oil and grease. It is logical to assume that such thin layers of oily film affect the adhesional characteristics of the fiber surface.

For cotton there is also the question as to the effect on soiling of the natural waxes on the fiber surface. In many fabrics no effort is made to remove this material, and even in those that do receive a kiering the wax is not completely removed by the ordinary commercial finishing operation. In addition, oils and fats in one form or another are frequently applied in the finishing process, a factor which should be taken into consideration in those uses where soil resistance is important and cleaning difficult.

Service conditions are of primary importance in determining the rate of soiling, the kind of dirt, and its location on the fabric. With this and an understanding of fabric and fiber characteristics, it is possible to obtain a reasonably good picture of the mechanisms most likely to be involved for any particular end use. Table IX gives an analysis of a variety of fabrics and end uses to show the most probable soiling mechanisms involved in each.

III. Methods of Measurement

To be complete, any method for evaluating the soiling and soil-resistant characteristics of a textile material must include three essential elements:

1. A means of soiling involving either a natural or synthetic soil applied by either a natural or artificial means of impingement.

2. A means of soil removal corresponding to the level of soil resistance sought and related to the conditions of maintenance of the fabric while in service. (For instance, in the case of rugs and carpets this may correspond to a vacuum cleaning; for draperies or upholstery to a simple brushing, shaking, or wiping of the surface; and for such frequently washed items as hand towels, to a laundering procedure.)

3. A means of measuring soil retention either by optically evaluating its visual effect or by determining the quantity present through chemical or physical analysis.

Some of the first attempts to measure the soiling characteristics of cotton and to evaluate the effects of various treatments were primarily aimed at improving launderability and were strongly influenced by the large volume of work that has been done in the field of detergency evaluation. While the measurement of detergency has a different goal in view, the two have much in common and the methods used are of interest as background for studying the measurement of soiling.

Detergency Evaluation

The usual detergency-evaluation test uses a soiled fabric having a degree of colored dirt retention high enough so that even the most efficient washing action does not completely remove the soil. This is felt necessary in order to obtain a ranking of detergent action. Thus, most soiled fabrics used for this purpose receive a higher level of discoloration than is ordinarily found in naturally soiled articles. An almost endless variety of synthesized soils and methods of application have been investigated. Vegetable, animal, and mineral oils or fats, together with a colored pigment, usually some form of carbon black, suspended in an oil solvent such as carbon tetrachloride, are typical of the synthetic soil mixtures employed. They are applied to the fabric by padding, brushing, or printing followed by evaporation of the solvent. To test the effectiveness of a detergent, the soiled fabric is laundered under controlled conditions; and a comparison of the reflectance readings before and after used as a measure of soil removal.

While considerable effort has gone into the selection of the oily constituents to insure their stability and similarity to natural dirt, there has been a notable lack of consideration for the chemical and physical characteristics of the colored component. In fact, the latter is often used only to furnish an optical

means of measuring the removal of the oils which are often considered the major factor in binding solid particles. Too, the method of applying the soil ignores the influence on dirt retention of the common mechanisms of impingement encountered in normal service. While such artificially produced soiling is frequently able to give a reproducible ranking of detergency, its correlation with actual service tests is often poor [31*A*, 98].

Utermohlen [115] found that artificial pigment soils were retained as tenaciously by cotton fabrics in the absence as in the presence of oily soils and concluded that the removal of oily films and solid particles from cotton are largely independent processes and that a pigment soil does not depend for its removal on the presence of an oil. He also introduced the use of iron oxide pigment as a soil, making it possible to follow by chemical analysis the colored solids content of the artificially soiled fabric. This technique allows the visual effect, as measured by reflectance, to be checked against the actual weight of solids present in the fabric. Although no simple relationship exists between reflectance and solids content, particularly in the low ranges encountered in normal soiling, mathematical expressions relating reflectance to artificial soil content have been developed [92].

Rotary-Disk Method

Because of dissatisfaction with the usual means of producing artificially soiled fabrics for detergency evaluation, Sanders and Lambert [98] devised a dry soiling technique using a modification of the Schiefer Abrasion Meter as shown in Figure 15. A weighed quantity of a synthetic soil designed to duplicate the composition of natural street dirt (see Table IV) is sprinkled on the fabric which is placed over the small 3-in. bottom disk. The upper disk with a surface of frosted glass is then lowered into contact with the fabric. A small circular soiled area is thus produced on the fabric by the rubbing action of the two disks which rotate in the same direction at the same angular velocity. It is interesting to note that soil retention was found to increase with the addition of moisture. Although the method was developed for detergency evaluations, the principle, which merits further consideration, has been applied by others to the study of soiling [21].

Chopped-Fiber Method

Compton and Hart [22] employed a novel method [90] which proved useful in studying the fundamentals of soil attachment to cotton fibers. It consists of chopping fibers into lengths of 1 mm or less, soiling by mixing with aqueous dispersions of carbon black, decanting with water, and filtering to obtain a pad of short soiled fibers. After drying of the fibers, the amount of soiling is measured by reflectance. The degree of soil retention may be evaluated by beating the fibers in water or in various detergents prior to filtering and drying. The method is best suited to the study of soiling characteristics of fibers since chopping eliminates the variables of yarn and fabric structure.

Particle-Count Method

As might be expected, the manufacturers of rugs and carpets were among the first to recognize the importance of a means for evaluating the drysoiling characteristics of their product. Although floor

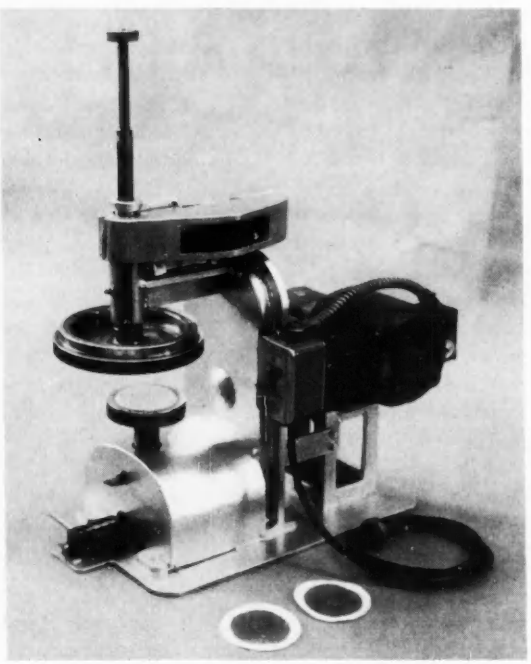


Fig. 15. Fabric soiling device developed by Sanders and Lambert from modification of a Schiefer Abrasion Meter

service tests and visual or photometric methods for evaluation of soiling are most often used in this industry, Masland in 1939 described an artificial method of soiling and a quantitative method of measuring soil retention [78, 79].

The soiling method consists of tumbling together in a ball mill for 1 hr a known weight of loose fibers and vacuum cleaner dirt. The fibers are then removed and placed in a container with a fine mesh screen top and bottom and the loose dirt is blown off with air. Basically similar methods were also used by Utermohlen [116] in a dry-soiling study of cotton fabric and by Fortess [37] for rug-pile fibers.

Soil retention is measured quantitatively by a particle-count method. A known weight of artificially soiled fibers, or pile fibers taken from a naturally soiled carpet that has been thoroughly vacuum cleaned, are scoured in an aqueous detergent solution. By means of a counting chamber mounted on a microscope, the dirt particles in a known volume of scouring liquor are counted. The number of particles removed per gram of fiber is calculated and used as a measure of soil retention.

Blower Test

This is one of three methods developed and proposed in a standardized form by the New York Section of AATCC [85] after a thorough review of the literature in this field.

It consists of passing equal volumes of air directly through the fabric samples being tested until the degree of soiling produced by the natural air-borne dust is enough to be evaluated visually or photometrically. Figure 16 illustrates the apparatus developed for this test. The fabric to be tested is mounted over the intake end of one of the tubes, each of which contains a motor and fan assembly. A means of individually controlling the air velocities through each of the tubes and thereby the volume of air passing through each of the fabrics is provided.

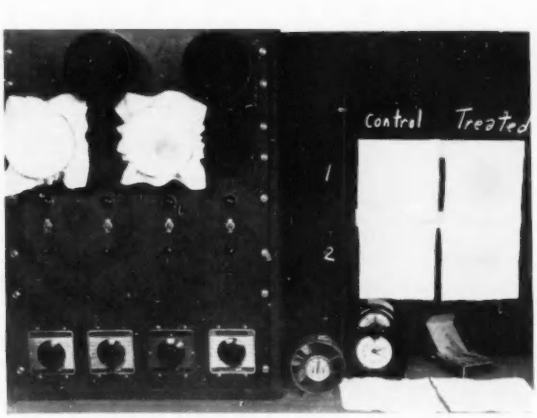


Fig. 16. Equipment and apparatus used in blower test.

The method is best suited for testing of fabrics used under service conditions in which the mechanisms of air filtration are the dominant means of soil impingement. Curtains hanging at an open window are typical of such conditions. The test is limited to those materials having an appreciable air permeability since the fabric acts as a filter medium in retaining particles that impinge from the moving air stream. Also, as the method is relative rather than absolute, it can be used only to compare the characteristics of fabrics tested simultaneously. The time required to reach a visible level of soiling depends on the concentration of particles and the volume of air filtered. In industrial atmospheres a 4-hr test period is said to produce the desired effect.

The method was shown to be quite sensitive to fabric construction and pore size which, in fact, was found to be of greater significance than fiber type. Through the use of standardized atmospheres, using artificially generated soil particles, it was suggested that the blower test provides a means of studying soil types and electrostatic effects. A basically similar method was described by Henno and Jouhet [46].

Floor Soiling Test

This is the second of the methods proposed by the AATCC committee and is a standardized form of the methods used by most carpet manufacturers in evaluating the soiling characteristics of their product. It is a service test dependent on natural dirt and foot traffic, and is thus limited to samples run simultaneously.

The method consists of inserting squares of test fabric into spaces cut out of a small rug as illustrated in Figure 17. The rug containing the test pieces is then exposed to foot traffic in such a way that all samples receive equal exposure. During the course of the test the rug is cleaned regularly by vacuum cleaning. When the discoloration has progressed to a visible degree, the samples are removed and compared visually or photometrically. The floor serv-

ice test is obviously limited to fabrics such as rugs and carpets where soiling is largely a function of the direct contact and vigorous mechanical action of foot traffic. Its greatest drawback is in the length of time (5 to 20 days in an industrial area) needed to reach a satisfactory level of soiling. However, since it duplicates not only the conditions but also the results of actual service it has been suggested as a means of checking the results of other methods to determine their correlation with actual service. Figure 18 shows a comparison of several rug samples exposed to soiling by this method. The degree of soiling should be judged by the figure given rather than by the appearance in the photograph.

Tumbler Test

This is the third method proposed by the AATCC committee and is a variation of similar methods previously described.

Test fabrics are tumbled end-over-end in a jar in a Launderometer together with a dry pigment soil and steel balls. After a short run, the test fabrics are removed, given a light shaking and placed in a perforated metal dusting cage with steel balls and again tumbled in the Launderometer to remove excess soil. The equipment and materials, except for the Launderometer, are shown in Figure 19. A synthetic soil is made by grinding together 5 parts of powdered bone charcoal and 50 parts powdered, calcined sodium sulfate. Thus the degree of soil retention can be measured visually or photometrically by reflectance, or quantitatively by analysis with barium chloride of the sodium sulfate remaining in the fabric.

The mechanisms of impingement present in the tumbler test are thought to be similar, although less vigorous, to those involved in the floor soiling method. Reasonably good correlation between the two has been reported for pile fabrics. As compared with the service test, the tumbler method has the advantage of greater speed and better reproducibility. It may also be adaptable to a wider range of fabric types than either the floor service or blower test.

Collar Test

A recent paper by the Washington Section, AATCC, describes a means of evaluating the soiling of fabrics in contact with the skin [123*A*]. The method employs a specially designed collar made from the fabric to be tested which is worn underneath the regular shirt collar. The test fabric, in contact with the neck over a period of time, accumulates soil at approximately the same rate and in a similar manner as an ordinary shirt collar. The rate and degree of soiling, or soil removal after cleaning, are indicated by changes in reflectance and color which are measured with a specially adjusted tristimulus colorimeter.

Since the method is dependent on natural service conditions, experiments must be carefully designed to be statistically reliable. Like other service tests, its main drawback is in the long period of time and the number of test subjects needed to complete a study. It should prove to be a valuable tool, however, for investigating this type soiling since it duplicates actual service conditions. It has already helped to point out the importance of yellowing as a factor in natural soiling [45].

IV. Soil-Resistant Treatments

Although few treatments are applied to cotton solely for the purpose of rendering the fabric more soil resistant, many do have some effect on this property. The stain-resistant qualities of certain finishes, frequently advertised, are normally only a side effect of their primary purpose of imparting water repellency. Similarly, such treatments as mercerizing or creaseproofing, which also show some tendency to reduce dry soiling, are not primarily used for that purpose.

The following discussion is an effort to bring together what little is known about the soil-resistant

Fig. 17. Inserting samples for floor soiling test.

characteristics of the various treatments applicable to cotton. Because of the limited information available, it is impossible to draw categorical or quantitative conclusions concerning them; and in many cases, even a qualitative evalution of their effect is lacking. In fact, even the soiling characteristics of cotton as compared to other fibers over a range of service conditions are far from being well established.

Fabric Construction and Design

Color. The application of color to textile materials is undoubtedly the oldest form of soil-resistant treatment known. Although its primary function is to enhance the beauty of the material, the action of color and color pattern in obscuring the effects of dirt should not be underestimated. Many apparel items and household fabrics are chosen with the idea that they will show soil less readily than a similar material of different color and pattern. While it cannot lower the quantity of dirt retained by a fabric or improve the ease of removal, color and pattern correctly used can serve to hide the effects of soil. In most uses where soil resistance is desired by the consumer, it is the visual effect rather than the actual quantity of dirt that is important.

A minute quantity of dirt affects fabric appearance far out of proportion to its weight, usually through a change in color or luster. For materials requiring a maximum of resistance to such change, proper selection of hue, color depth, and design, can be of considerable help. Large, irregular patterns are often a more effective means of camouflaging spots or stains than repeated small figures where the eye is immediately attracted to any change in the overall regularity. In selecting the most suitable colors for soil resistance, the type of dirt and the conditions to be encountered during use must also be considered. In laundering, "blueing" or blue fluorescent dyes are commonly used to neutralize the residual yellowness of a fabric and enhance its appearance of whiteness. In general, dark patterns hide soil better than white or pastel colors, but neutral colors in medium depths are sometimes effective. For instance, light-colored particles, such as lint or cigarette ash, are more easily seen against a dark background.

Yarn and fabric structure also play an important part in the soiling characteristics of textile materials. However, from the lack of information on the subject, it is evident that very little organized effort has as yet been made to study this means of improving serviceability. Here again, knowledge of service conditions is all-important in helping to determine optimum design and construction.

Fabrics coated with plastic films and materials made of coarse monofilament yarns show a consid-

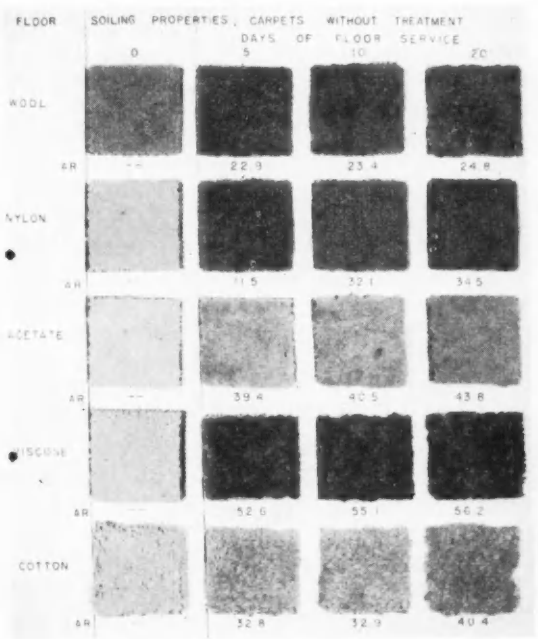


Fig. 18. Comparison of soiling of rug samples made from various fibers tested by floor service method. ΔR = change in reflectance.

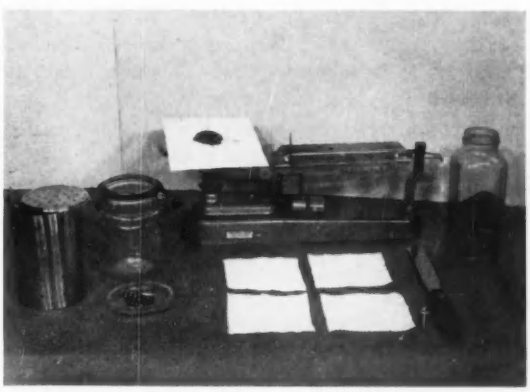


Fig. 19. Equipment and materials used in tumbler test.

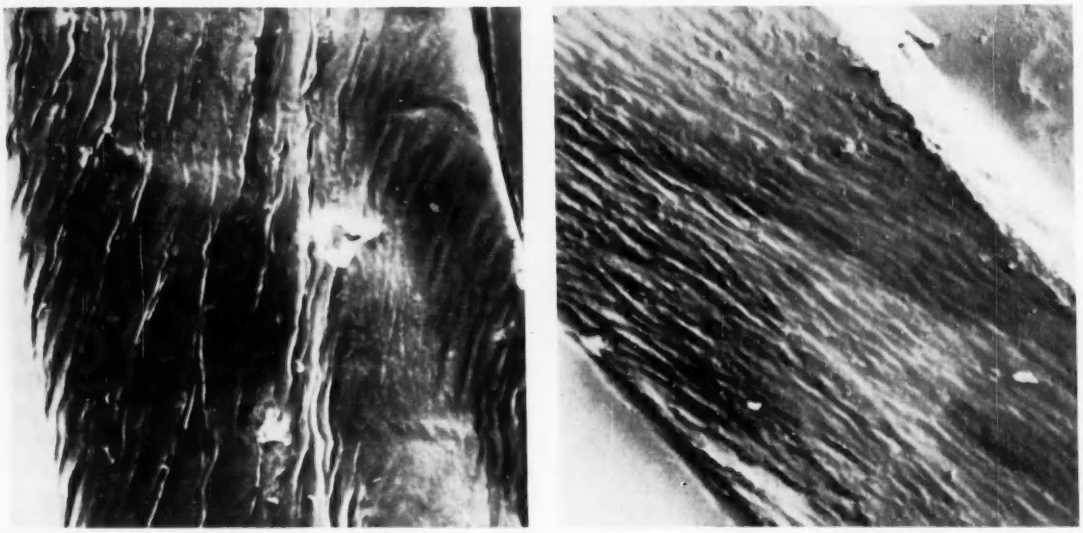


Fig. 20. Electron micrographs of replicas of cotton fiber surfaces, ×8000 approximately. *A*, An untreated fiber; *B*, a mercerized fiber.

erable degree of soil resistance because of the inherent absence of some of the factors of mechanical entrapment. In such materials, however, many of the most desirable characteristics of a textile fabric are lacking—those qualities dependent on the complicated interyarn and interfiber space systems present in most textile structures.

With an understanding of the fundamentals of soiling and a knowledge of the conditions to which a material will be exposed in service, the designer can manipulate fabric structure within certain limits to provide improved soil resistance.

Chemical Modification

Mercerization was one of the earliest chemical treatments to be applied to cotton. As a finish its main purpose is to improve luster, although it also makes cotton stronger, more absorbent, and easier to dye. Comparative tests [85, 116] show that it also improves soil resistance. Cotton mercerized prior to dyeing has less tendency to "stain" in the presence of acetate dyestuffs which are dispersions of fine particles [43].

Although significant, the improvement resulting from mercerization is by no means a final solution to the problem of soiling. While its influence on this property alone is seldom sufficient to warrant the additional cost of the treatment, the fact that it does have an effect justifies a careful scrutiny of the means by which the change is brought about.

The change in shape and the increased size and smoothness due to permanent swelling is generally agreed to be the reason for its improved soiling characteristic. Microscopic examination shows that mercerization either with or without tension produces a smoother, rounder fiber. Since it eliminates most of the larger surface irregularities present as convolutions in the raw fiber, there is less surface area and fewer gross irregularities for dirt entrapment. It also has an effect on the fine surface structure as can be seen from a comparison of the electron micrographs in Figure 20. Figures 21 and 22 show the change in cross-sectional outline produced by mercerization; while Figure 23 shows the longitudinal shape as compared with the untreated fiber shown in Figure 7.

Partial acetylation and carboxymethylation of cotton have also been shown to improve its soiling characteristics [26, 116]. Effects similar to those of mercerization might be expected, since these treatments also produce permanent swelling. However, room for doubt exists as to the exact mechanisms involved since aminoethylated cotton, which also swells the fiber, did not show, in one case at least, the same improvement produced by the other treatments. This difference between carboxymethylated

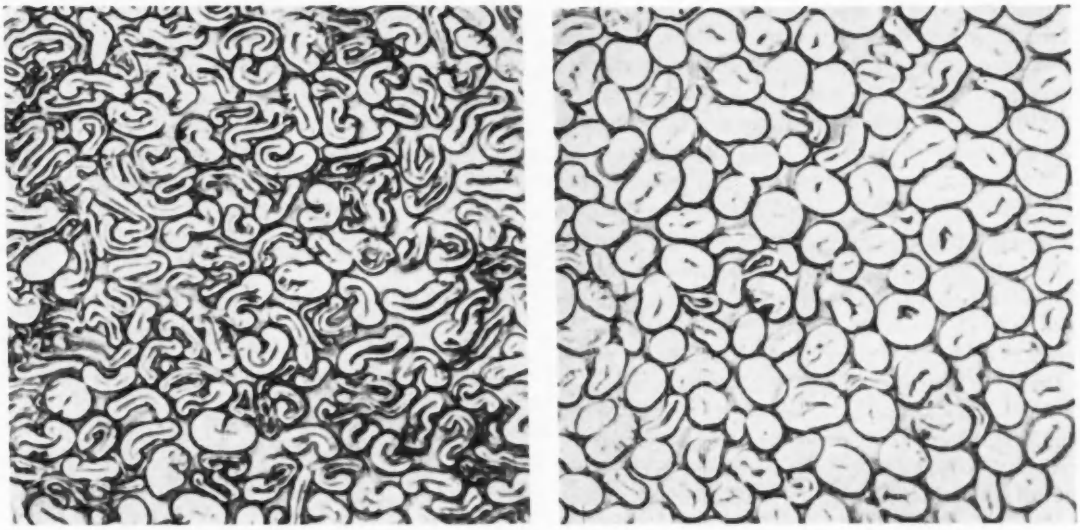


Fig. 21. Photomicrographs of cotton cross section, ×500. *A*, Untreated; *B*, mercerized without tension.

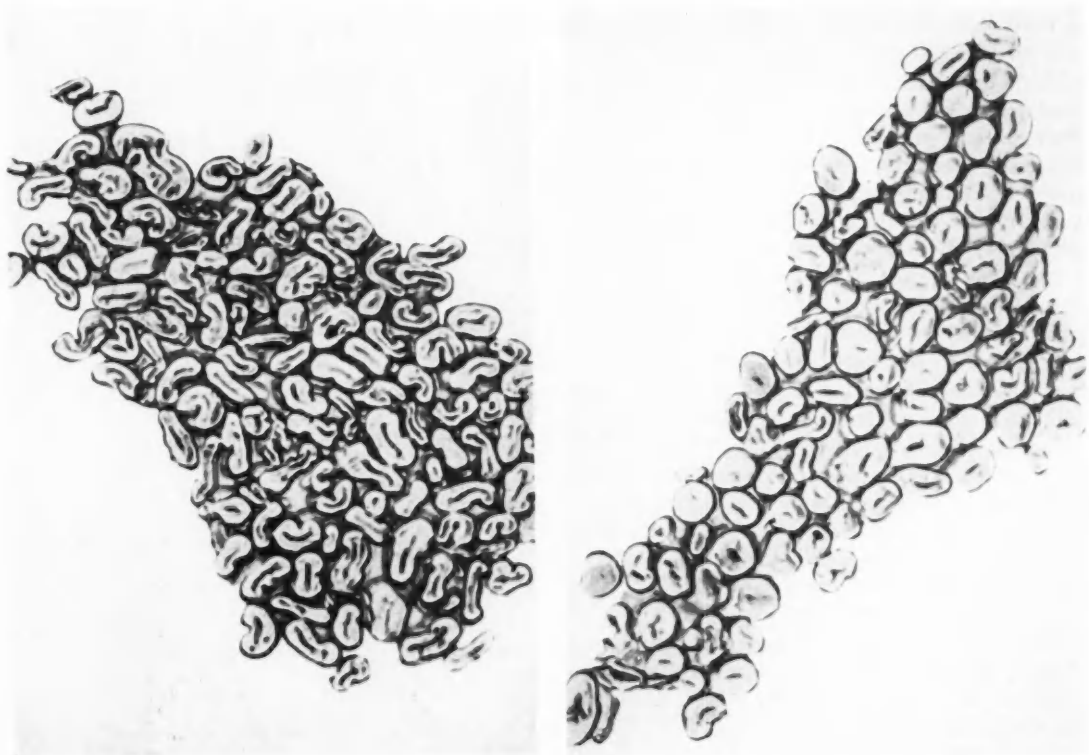


Fig. 22. Cross sections of cotton yarn, ×500. *A*, Unmercerized; *B*, mercerized at normal length

and aminoethylated fabrics requires further study. Since these two treatments are thought to impart opposite electric charges, it was one of the factors leading to the suggestion [116] that treatments which can confer cation exchange properties to cotton cellulose affect soil retention.

Additive Treatments

Starch was probably one of the first additive finishes to be applied to cotton. It was also one of the earliest to be investigated for its influence on soiling [64, 111, 116]. It is not very effective as a soil-resistant treatment although 5% or more on a fabric is known to improve the ease of dirt removal in laundering. This action is probably the result of its film-forming action in preventing the deeper penetration of soil by filling the fabric pores [123*A*].

Carboxymethylcellulose. The effectiveness of sodium carboxymethylcellulose (CMC) in detergent mixtures in preventing redeposition during laundering has been recognized for some time. A low-substituted, alkali-soluble form has been marketed as a soil-resistant finish for cotton. CMC has also been promoted as a replacement for starch in household and commercial laundry sizing. A more recent development [106] has been the suggestion of adding small amounts to the final rinse water after each laundering where as little as 0.25% is said to improve soil resistance and aid removal in subsequent laundering without changing the appearance or hand of the fabric.

Two different theories are advanced to explain the action of CMC. One [54, 89, 124] suggests that its action is due to an increase in the electrostatic repulsive force that normally exists between the negatively charged soil and the negatively charged fiber. In the case of the pretreatment of fabric, the negative charge on the fiber is increased; while as a component of the detergent bath it increases the negative charge on both soil and fiber. The mechanical theory suggests that a thin film of CMC on the fiber surface physically blocks the entrance of small dirt particles; and that, when present in the detergent, its long-chain molecules become adsorbed on the surfaces of the soil particles, thereby increasing their effective size to the point where they are too large to become mechanically entrapped in the microscopic fiber surface irregularities.

Although the synergistic action of CMC in detergency is well established, its effectiveness as a soil-resistant treatment for cotton fabric has not as yet been fully evaluated. Figure 24 illustrates the result of an experiment showing improvement in the soiling characteristics of a treated cotton rug sample. Table X compares the action of CMC with other water-soluble film-forming materials as an aid to soil removal in laundering [55].

Water-repellent treatments. By preventing fiber swelling and absorption of the liquid into the fabric

Fig. 23. Longitudinal view of mercerized cotton fiber, ×500 (cf. unmercerized fiber, Figure 7).

capillaries, water-repellent treatments provide a considerable degree of protection against water-borne dirt and stains, provided the liquid soil is quickly removed and not allowed to remain in contact with the fabric for too long a period. The nonpermanent-type finishes, however, employing waxes or metallic soaps, furnish no protection to the fabric against greasy or oily dirt and, in fact, tend to increase the retention of dry soil through the mechanism of oil bonding [85].

Plastic coatings. Some of the earliest references to soil-resistant treatments relate to patents obtained for improving the washability of cotton fabrics by coating with nitrocellulosic lacquers [17, 131]. Fabrics coated with vinyl or other thermoplastic films are common today. Since dirt retention is limited to a single plain surface, removal by brushing or by wiping with a damp cloth is a simple matter. Plastic films impart stiffness and other completely different characteristics to the fabric and thus limit the uses to which this form of soil-resistant treatment can be applied.

Crease-resistant finishes. Improved soiling characteristics have been reported for several of the crease-resistant and fabric-stabilizing finishes [25, 85]. However, the mechanism by which urea and melamine formaldehyde type resins and crosslinking materials like glyoxal, when condensed within the fiber, reduce soil retention requires further study. Whether it is accomplished by reducing swelling, by a change in fiber surface characteristics, or by some surface energy function, is not clear.

Presoiling treatments. The most recent development in the field of soil-resistant finishing has been the introduction of methods for presoiling a fabric with very finely divided colorless particles. There is as yet no sound basis for judging the degree of soil resistance obtainable by this method since little experimental data have been released and too short a time has elapsed for practical evaluation. While to date this method has been used almost exclusively for improving the soiling characteristics of pile fabrics, particularly rugs and carpets, there is reason to believe that the principle can also be applied to other uses where similar mechanisms of soiling are involved.

Under normal service conditions where rugs are exposed to foot traffic and to periodic vacuum cleaning it is common experience to find discoloration eventually reaching a plateau or point beyond which little further change in appearance takes place with continued service and cleaning. (See Figure 18.) Available sites on the fiber surfaces become gradually filled, and subsequent accumulations of dirt, having fewer and fewer places to occupy, become

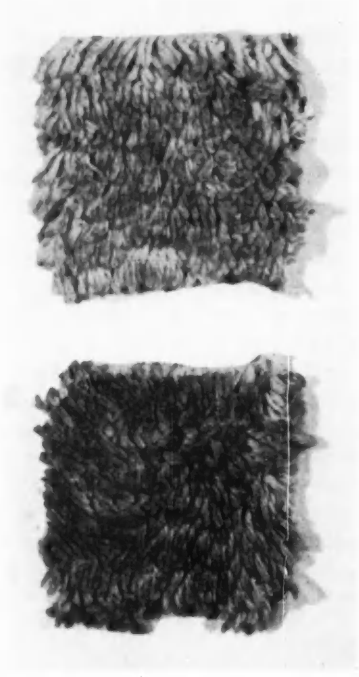


Fig. 24. Cotton rug sample treated with CMC and exposed to floor service test. *Top*, CMC treated; *bottom*, untreated control. The change in reflectance of the treated rug was 64% of that of the untreated.

TABLE X. Comparison of CMC and Other Water-Soluble Film Formers *

Sizing Material	Add-On (%)	Soiled Reflectance (Hunter)	Washed Reflectance (Hunter)	Whiteness Retention (%)
None	0.00	30.4	35.2	82.1
CMC (medium viscosity)	0.09	34.0	47.4	100.0
Sodium alginate	0.09	30.4	37.9	87.9
Starch (proprietary liquid)	0.09	23.7	31.7	67.0
Starch (thick boiling corn)	0.10	23.9	30.9	69.4
Starch (modified)	0.09	27.5	35.2	89.0
Polyvinyl alcohol	0.08	29.1	35.6	88.1
Polyvinyl acetate	0.10	23.6	29.8	71.5

* Source: *Chemical and Engineering News* [55].

progressively easier to remove until, theoretically, the amount removed by periodic vacuuming equals the amount impinging on the rug.

The principle involved in the presoiling method is to reach this soil saturation level by applying in the finishing operation a white or colorless "dirt" to occupy the available sites before the fabric is exposed to service. The artificial "soil" must have a particle size range at least as fine, if not finer, than that of the natural dirt to be encountered in service and should presumably be attached to the fibers in a similar manner to natural soil.

A recent patent [19] describes the use of commercially available colloidal suspensions of silica or clay ranging in size from 0.025 to 10 μ. Applications of between 0.25 and 0.5 oz/sq yd, to the rug pile by spraying or brushing followed by drying, are recommended. Small amounts of colored material may also be added to overcome the tendency of the white powder to lighten the color of dyed fabrics.

It is interesting to note that the same principle has been used for many years in other fields. For instance, in pastry making, flour is sprinkled on a board to prevent the sticking of the dough; and in the working of putty for window glazing, its stickiness is overcome by rubbing powdered chalk into the hands. The principle of cleaning with finely divided particles has also been known for some time, as in the use of oiled sawdust in floor-sweeping compounds, fuller's earth for removal of grease and oil during woolen finishing, and wood flour and clay together with small amounts of organic solvents in rug-cleaning powders.

V. Opportunities for Further Research

Only in the past few years has the consumer even begun to appreciate the inherent possibilities in improved soiling and cleaning characteristics for textile materials. In part, this growing awareness of the problem stems from the promotional effort that has accompanied the introduction of the newer synthetic fibers. While these too have soiling problems peculiar to themselves, the fact remains that a desire for this quality is being aroused. Active demand already exists in large markets although in a restricted number of items. As improvements are made, increasing demand is certain to develop in other markets where only passive interest now exists.

The value of the soil-resistant properties of a fabric is generally appreciated by the consumer only after a period of service. It is a factor not easily recognized at the time of purchase, and, unlike such qualities as design, luster, or crease resistance, has had little sales promotion and far less research attention. It is thus subject to all the possibilities of new and unexplored fields. In such neglected areas empirical or trial-and-error methods often prove rewarding. On the other hand, the fundamental approach should not be neglected since only by this means can long-range progress be assured.

Throughout the preceding discussion, unanswered questions are raised and suggestions made concerning the need for further work. These, together with the following, are in no way meant to limit the thinking or restrict the approach to future research. To those now engaged in actual studies in this field, other and even more productive ways of attacking the problem will become apparent, particularly as a greater body of organized information is accumulated on this comparatively new subject.

1. *Fiber Selection and Breeding*

The cotton fiber is known to vary widely, according to variety, in such shape factors as size, circularity, and spirality. They may also differ significantly in fine surface structure. Information on these factors should be accumulated and their effect on dirt retention investigated. Cottons in a range from coarse to fine should be compared by spinning and weaving into a series of comparable yarns and fabrics which should then be evaluated over a wide range of service conditions. If significantly different soiling characteristics are shown by any particular type, efforts should be made to expand the desirable characteristics by breeding.

Heretofore, the efforts of most cotton breeders have been largely directed toward producing cottons with long, fine fibers for maximum strength. However, large markets exist in which fineness and strength are of only secondary importance. In the case of pile yarns for cotton carpeting, for instance, soil resistance and crush resistance are of major interest, and it is likely that coarser-fibered cottons could improve both these characteristics.

2. *Test Methods*

At the present stage in the development of soil resistance, the importance of adequate test methods can hardly be overemphasized. Service tests that

reproduce the effect because they employ all the conditions of natural soiling are still the only dependable means of comparing and evaluating the effectiveness of treatments. Their usefulness is limited because of the time involved in soiling and the fact that, since all variables are not subject to control, only those samples run simultaneously may be compared directly. While they serve as valuable means for checking other tests, efforts should be continued toward finding fast, reproducible laboratory methods that correlate with service.

One step should be a more thorough study of the chemical and physical composition of natural dirt, particularly that portion still adhering to the fabric after mechanical cleaning. Such a study should be directed toward the development of representative laboratory soiling mixtures. An approach to the problem of determining the particle-size range of the dirt retained by fabrics and fibers would be to apply synthetic soils having a predetermined particle-size distribution, remove the excess by mechanical agitation, and then measure the size distribution of the remaining soil not picked up by the material. A change in the shape of the distribution curve would indicate that selectivity had taken place within a certain particle-size range. Both the light and electron microscopes should prove to be valuable tools in the study of particle size, size distribution, and the location of dirt on the fiber.

The question should be answered as to correct interpretation of relative and absolute methods of measurement as given by reflectance readings on the one hand and quantitative chemical analysis of soil content on the other. There is little doubt that the layman is primarily interested in appearance involving the change in hue as well as in reflectance caused by soil. A technician, however, appreciates the fact that scientific improvements often result from working with absolute quantities. While neither approach should be neglected, appearance must be the final criterion. The use of radioactive tracer materials as a component of soiling mixtures, which so far have been used only to study detergency, may provide a means of investigating this phase of the subject [8, 66, 80].

3. *Chemical Modification of the Fiber*

Mercerization, partial acetylation, and partial carboxymethylation have been shown to improve cotton's soil resistance. Other treatments, such as aminoethylation, are less effective. Such differences require further study. Other chemical modifications of the fiber should also be evaluated and the treatments that reduce soil retention should be carefully examined to determine the mechanisms involved.

Too little is known about the action of various chemical agents on the fine surface structure of the fiber. That chemical action can produce a change in this area is illustrated by Figure 25, an electron micrograph of a cotton fiber surface treated for 32 sec with 66% sulfuric acid. This can be compared with an untreated fiber shown in Figure 9. While the light microscope is capable of disclosing the relatively coarse surface effects, the electron microscope should prove valuable in determining the effect on fine surface structure of chemical modifications and surface treatments.

4. *Additive Finishes*

Of the numerous treatments applicable to cotton fabrics, the effect on dirt retention and ease of removal of only a few are known, and these only quali-

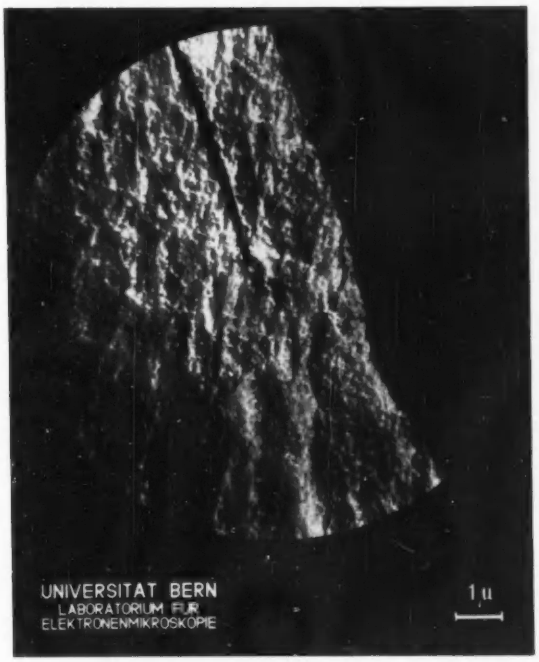


Fig. 25. Electron micrograph of cotton fiber surface showing effect of treatment with strong sulfuric acid, ×7000.

tatively. In general, finishes employing oils and waxes increase soiling through oil bonding. However, a recent series of experiments, involving a variety of softeners and lubricants used in the manufacturing of acetate pile carpeting, show that certain antistatic wax finishes do not cause increased soiling [37]. A similar survey of the compounds commonly used in the finishing of cotton fabrics should be made to determine their effect on soiling. The influence of the naturally occuring wax located on the surface of the raw cotton fiber should also be included in such a study.

Additive treatments offer many possibilities for the improvement of fabric soiling characteristics. Water-repellent finishes offer protection against water-borne dirt and stains, but many tend to increase dry soiling. Efforts should be directed toward finding water-repellent finishes that overcome this tendency. Silicone treatments [27, 28, 30, 120] are reported to have improved soiling characteristics in respect to oily dirt, and when applied to paper in combination with water-soluble cellulose ethers are able to reduce the adhesion which normally exists with such sticky substances as asphalt and rubber [29].

The oil-resistant properties of new and interesting compounds warrant further investigation. One of these, perfluorodecanoic acid, is said to impart a high degree of nonwettability when applied in very thin layers to polished surfaces [5, 101]. On such surfaces, liquids of all types including hydrocarbons show a high contact angle and will not spread. The presence of the CF_3 group on the end of a straight-chain acid molecule is believed to be responsible for the lowered attraction for liquids. The fact that surface roughness greatly impairs this property, especially with liquids of low surface tension, would appear to limit its use on fabric surfaces. However, the action of this and similar substances on fiber surfaces should be investigated as a means of reducing the spreading and adhesion of oily soils.

Coating with vinyl or other plastic films prevents penetration of dirt into the fabric. It is possible that the same principle can be applied to fiber surfaces. A process that could coat individual fibers without bonding them together as a continuous film would prevent occlusion of fine soil particles by the fiber surface while maintaining other desirable fabric characteristics. This might be possible through the use of cuprammonium or zinc chloride treatments.

The mechanisms by which several of the crease-proofing resins improve soil resistance should also be determined. Such a study should include an investigation of fiber surfaces and the effects of changes in the swelling and stress-strain properties of the fiber.

The possibility of extending the presoiling type of treatments to fabrics other than rugs and carpets should be investigated.

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It should not be inferred that the technical opinions expressed in this report are necessarily those of the authorities listed above.

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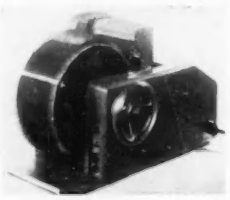
Manuscript received September 27, 1954.

MECHANICAL COTTON BLENDER

"A time saver for effective blending and sampling"



The redesigned blender was a development of the Cotton Branch, Production and Marketing Administration, U. S. Department of Agriculture, Washington, D. C. Effective blending of cotton samples is now assured with this new machine which will handle 10 gram samples. This method takes about one hour to prepare 8 samples. Hand methods take 20 times as long for comparable weight samples.

Mechanically blended cotton samples weighing approximately 10 grams will provide a suitable source of test specimens for the following laboratory fiber tests:— Array length, fineness and maturity, Pressley strength, Fibrograph length, Micronaire fineness, microprojector maturity, and Nep count in raw cotton. The blender can be used also to blend subsamples into a composite bat to represent a raw stock mix for the source of laboratory test specimens.

Reference: ASTM Designation D 1441-54, "Sampling Cotton Fibers for Testing."

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Hotel Commodore New York

March 10–11, 1955

This, the Twenty-Fifth Annual Meeting of TEXTILE RESEARCH INSTITUTE marks the twenty-fifth anniversary of the founding of the Institute, and a program of exceptional interest has been arranged.

All sessions and luncheons will be held in the Mail Ballroom of the Hotel Commodore.

The terms "Operations Research" and "Automation" have recently come into prominence. Speakers have been obtained who are especially well qualified to explain these latest developments and how they are being successfully applied in industry. Certain recent trends and developments in research will be described; and the broad problems involved in textile education will be discussed.

A feature of the two-day meeting will be a luncheon each day with a guest speaker widely known for his ability to give a stimulating address.

As in former years, in the West Ballroom some new developments in laboratory and control instruments as well as in new fibers and dyes will be shown.

Everyone who is connected in any way with the textile industry, its allied branches and suppliers is invited to attend and will find much of specific interest.

General Chairman

MILTON HARRIS

Chairman, T.R.I. 25th Annual Meeting Committee

Thursday, March 10, 1955

MORNING SESSION 10:00 A.M.

REVIEW OF T.R.I. RESEARCH IN 1954

J. H. DILLON

Director, Textile Research Institute

TEXTILE EDUCATION AT THE UNDERGRADUATE AND GRADUATE LEVELS

BERTRAND W. HAYWARD

Chairman, T.R.I. Education Committee

LUNCHEON 12:45 P.M.

Toastmaster, SYDNEY CONE

DON'T SELL COTTON SHORT

HUGH COMER, Guest Speaker

Board Chairman, Avondale Mills

AFTERNOON SESSION 2:15 P.M.

CHEMICAL STRUCTURE AND USEFUL PROPERTIES OF TEXTILES

ARNOLD M. SOOKNE

Assistant Director, Harris Research Laboratories

THE MECHANICAL BEHAVIOR OF FIBERS

ALEXANDER BROWN

Carbide and Carbon Chemicals Co.

Friday, March 11, 1955

MORNING SESSION, 10:00 A.M.

OPERATIONS RESEARCH

GLEN D. CAMP

Consultant to President, Melpar, Inc.

AUTOMATION

JOHN DIEBOLD

John Diebold and Associates, Inc.

LUNCHEON 12:45 P.M.

Toastmaster, PERCY HOWE

GEORGES F. DORIOT, Guest Speaker

BUSINESS MANAGEMENT AND THE FUTURE OF THE TEXTILE INDUSTRY

Professor of Industrial Management, Harvard Business School, Harvard University

AFTERNOON SESSION 2:15 P.M.

CONTROL OF CARPET SOILING

PATRICK FLORIO and EMORY MERSEREAU

Alexander Smith, Inc.

TEXTILES WITH NEW PROPERTIES FROM CELLULOSE TRIACETATE

R. G. STOLL

Assistant Manager, Summit Research Laboratories, Celanese Corporation of America

For list of exhibitors and products, see next page.

Exhibits*

C. A. Brinkman & Company
Great Neck, New York

Interference Microscope, which opens novel ways for investigations in fiber microscopy.

Analytical and Micro Balances, representing new advances in rapid weighing.

Photomicrographic Camera, with built-in exposure meter.

Micro Manipulator, for industrial work.

Brush Electronics Company
Cleveland, Ohio

Imperfection Counter, developed in cooperation with the Institute of Textile Technology. Accurately counts neps and other yarn imperfections at 100 yards per minute and totals them automatically.

Tension Analyzer, developed in cooperation with the Textile Research Department, American Viscose Corporation. Accurately measures and records actual tension levels and variations in filamentary material being processed.

Uniformity Analyzer, measures and permanently records irregularities in weight per unit length of yarn, roving, and sliver.

Celanese Corporation of America
Summit, New Jersey

Arnel, the new triacetate fiber. A variety of Arnel fabrics and garments illustrating a range of constructions, textures, and colors will be displayed. Various properties of Arnel will be demonstrated.

Custom Scientific Instruments
Arlington, New Jersey

High Speed Angle Recording Tensiometer, measures tensions in limited places. Can be inserted into running yarn systems. Novel technique for overcoming pickup problems.

Electrical Resistance Tester, evaluates tendencies of materials to accumulate and hold electrostatic charges.

Pilling Tester.

Mechanical Cotton Blender, for samples.

Eastman Chemical Products, Inc.
New York City

Chromspun-Dyed Fiber, and applications thereof.

Gas- and Light-Fast Dyes, on textiles.

White Estron, in commercial uses such as for filter cloths.

Fabric Development Tests
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Drape-Flex Stiffness Tester, improved model. Use has been incorporated into Standard Federal Test Specifications. Developed for U. S. Naval Supply Office.

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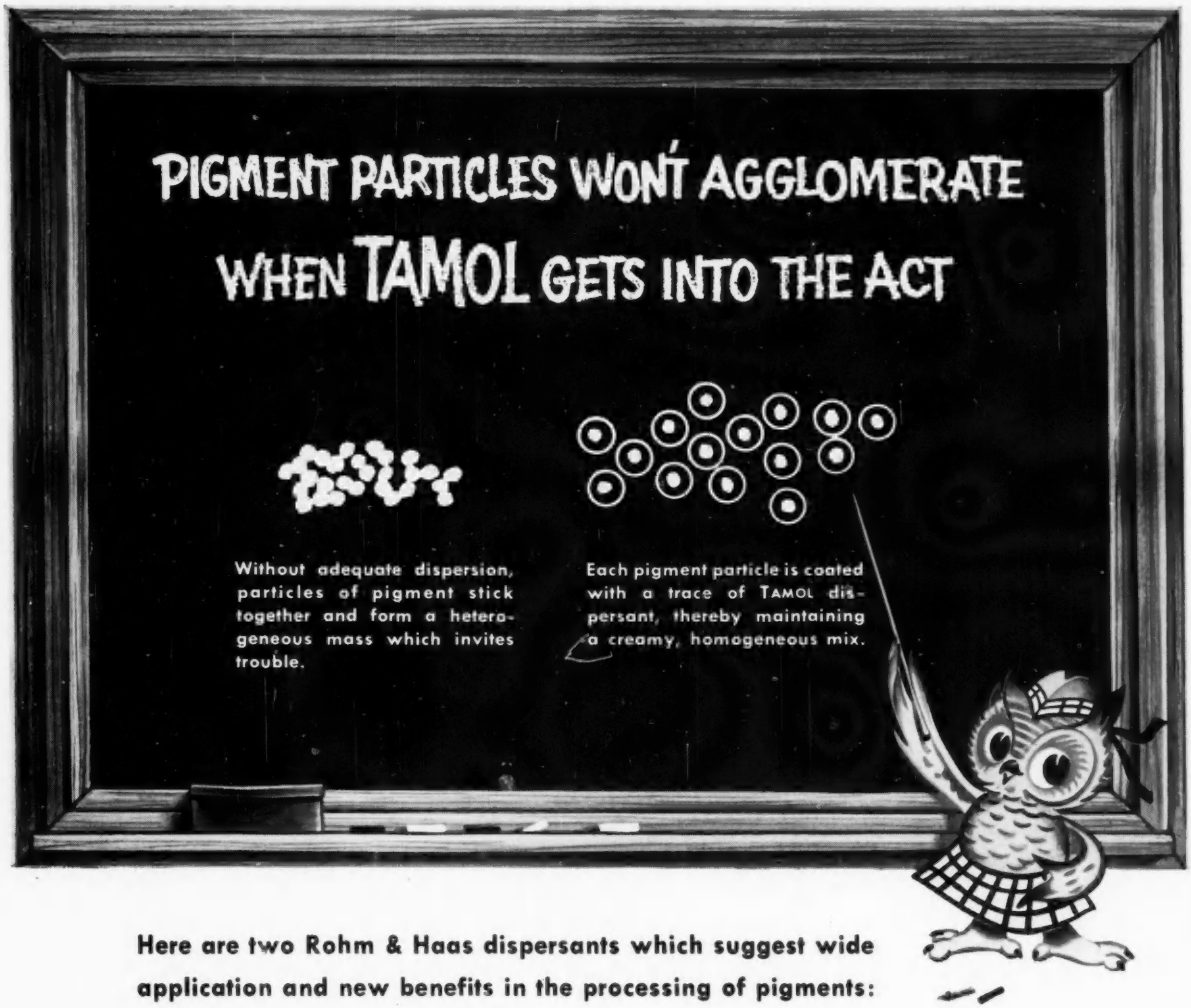
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